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DEVELOPMENT OF HIGH ENERGY DENSITY PRIMARY BATTERIES

SECOND QUARTERLY REPORT

by

S.G. Abens, T.X. Mahy, and W.C. Merz

22 September 1965 to 21 December 1965

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

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Studies of the CuF_2 -Li system for the development of high energy density primary batteries were conducted. The solubility of CuF_2 in propylene carbonate and butyrolactone increased with increasing water contamination levels. Pasted CuF_2 electrodes were discharged at 10 mA/cm^2 in 4.7M LiClO_4 -methyl formate electrolyte with reduction efficiencies of 70 percent. With 1.4 M LiClO_4 -propylene carbonate electrolyte, CuF_2 -Li cells showed about 80 percent capacity retention after four weeks stand at -15°C . Vacuum drying of CuF_2 before construction of cells improved capacity retention.

Author

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1. SUMMARY

Experimental work toward development of high energy density CuF_2 -Li primary cells was conducted. It is desired to evolve (a) batteries operating at the 1-10 hour discharge rate, and (b) batteries operating at the 100-1000 hour discharge rate. It is felt that the former type may be designed with reserve activation, while the latter must be constructed "wet" and, therefore, must have adequate wet shelf life. The experimental work was conducted in the following general areas of investigation:

Electrolyte Systems Studies

For use in the 100-1000 hour battery, 1.4M LiClO_4 -propylene carbonate electrolyte is considered to be of major interest, while 1.4M LiClO_4 -butyrolactone is being considered as a probable alternate choice. The effect of water contamination level on the stability (rate of discoloration) of lithium test strips in these electrolytes was studied. The effect of water contamination was shown to be detrimental, but the presence of LiClO_4 at the same water contamination level appeared to be improving the lithium metal stability.

Karl Fischer analysis of volume fractions of redistilled butyrolactone and propylene carbonate showed that the first 5-10 percent of the distillate is high in water content, indicating that a substantial reduction in water content can be achieved by rejecting the initial portion of the distillate.

The effect of water contamination on the solubility of CuF_2 in butyrolactone- LiClO_4 and propylene carbonate- LiClO_4 electrolytes was studied. The solubility of CuF_2 in both electrolytes increased with increasing water contamination over the range of 400 to 8000 ppm, the solubility being proportional to the initial water content of the electrolyte solution.

Positive Electrode Studies

Studies of thin-plate CuF_2 electrode construction for application in the 1-10 hour battery were conducted. Pasted plates having the dimensions 1.5 x 1.5 x 0.025" were discharged against lithium negative electrodes at 10 mA/cm² in 4.7M LiClO_4 -MF electrolyte. CuF_2 reduction efficiencies in the range of 60 to 70 percent were obtained in some cells at average cell potentials in excess of 2.7 volts. Typical electrode composition in this test series consisted of 100 parts of CuF_2 , 10 parts of graphite, and 1 part of cellulose acetate. The pasting solvent was a 10 percent methyl alcohol-90 percent ethyl acetate mixture.

The filter-mat CuF_2 electrode composition, which is presently used in test cells for the 100-1000 hour discharge range application, was studied by varying the CuF_2 -graphite-paper fiber ratio in the mat. Poor wetting of the electrode matrix with the 1.4 M LiClO_4 -propylene carbonate electrolyte was observed in the cells which had a high graphite-paper fiber ratio in the CuF_2 mix. This condition was believed to have affected the discharge efficiency in the affected cells (efficiency varied over the range of 40-80 percent at the 10-day rate). It is believed that vacuum impregnation of the electrodes should be employed in future comparative tests of this electrode construction.

Cell Systems Studies

A study of the rate of self-discharge in CuF_2 -Li cells with LiClO_4 -propylene carbonate electrolyte was initiated during the quarter. The cells had one filter-mat CuF_2 electrode and two lithium sheet electrodes. The water content in the electrolyte was about 500 ppm.

One group of cells was constructed with CuF_2 having a water content of 2.8 percent as determined by X-ray analysis. A second group of cells had the above CuF_2 dried for 16 hours at 70°C and 100-200 microns pressure. Test cells were discharged immediately after activation with the electrolyte solution, and after wet stand at $+35$, -15 , and -55°C .

At 35°C capacity retention was poor in all cells, but at -15 and -55°C , retention was in the order of 80 percent after four weeks of stand for the cells constructed with dried CuF_2 . Inspection of cells after stand and/or discharge showed green deposits on the lithium electrodes characteristic to copper compounds; this condition was more pronounced in the cells which had exhibited poor shelf life. Apparently, the solubility of CuF_2 limited the shelf life of the cells.

2. INTRODUCTION

The purpose of this program is the development of high energy density primary battery systems suitable for space flight applications. Two types of batteries, differing in energy density and discharge rate requirements, are being sought: 1) batteries capable of delivering 200 watt hours per pound at the 100-1000 hour discharge rate, and 2) batteries capable of delivering 50 watt hours per pound at the 1 hour discharge rate. For both types of batteries, the CuF_2 -Li couple is being considered. For the low-rate battery, a propylene carbonate-lithium perchlorate electrolyte system is of primary interest, while for the high rate system a methyl formate-lithium perchlorate electrolyte is being considered. These systems emerged as the most promising at the conclusion of two previous yearly contract periods (NAS 3-2775 and NAS 3-6004).

Development of a high efficiency CuF_2 electrode is not considered to be the main task at this time since the presently used construction has produced cathodic efficiencies in the order of 80 percent of theoretical (2 electron reduction). High energy-to-weight ratios have been demonstrated with both propylene carbonate and methyl formate electrolytes. The major remaining problem, therefore, appears to be the chemical instability of these systems; *i. e.*, lack of shelf life capability. While the 1-hour battery with methyl formate electrolyte could be designed for reserve activation, the low rate system must possess sufficient chemical stability to be useful at the 1000-hour rate.

The work described in the present report is a continuation of the activity reported in the First Quarterly Report (NASA CR-54859). For the high rate system (methyl formate electrolyte) thin pasted CuF_2 electrodes capable of discharge at the 1-10 hour rate were being developed. For the low rate (propylene carbonate electrolyte) battery, filter-mat CuF_2 electrodes were under development. The chemical stability of the CuF_2 and Li electrodes in electrolyte solutions was studied, and self-discharge tests with CuF_2 -Li cells having propylene carbonate electrolyte were performed.

3. DESCRIPTION OF EXPERIMENTAL WORK

3.1. ELECTROLYTE SYSTEMS STUDIES

3.1.1. Stability of Lithium in Butyrolactone and Propylene Carbonate

A criterion which has been continuously employed on this program for the evaluation of solvents and electrolyte solutions is the rate with which a lithium test strip is attacked when immersed in the liquid. A large number of these tests have been performed with materials as received from the manufacturer; i. e., the quality was assumed to be as stated by the manufacturer for the particular grade of material. This degree of control does not appear to be adequate for more exact studies of the role of impurities on the stability of the lithium, especially since the quality of the solvents is variable even though supplied from a single lot by the manufacturer, as can be seen from Tables I and II.

TABLE I

WATER CONTENT OF 500 gram CONTAINERS OF BUTYROLACTONE

<u>Container No.</u>	<u>Color</u>	<u>Water, ppm</u>
1	Pale Yellow	2050
2	Water White	434
3	Water White	390
4	Pale Yellow	2840
5	Pale Yellow	2940
6	Off White	1700
7	Water White	470
8	Water White	380

TABLE II

WATER CONTENT OF 500 gram CONTAINERS OF PROPYLENE CARBONATE

<u>Container No.</u>	<u>Water, ppm</u>
1	710
2	440
3	180
4	270
5	220
6	750

Thus, to assure uniformity of water contamination levels in the tests, the contents of every individual container must be dried and analyzed before use.

The effect of water contamination level on the stability of lithium samples in BL and PC, and in LiClO_4 solutions in the same solvents was observed by performing a comparative test on several materials at room temperature. Each of the two solvents was used with two water contamination levels (2000 and 390 ppm for butyrolactone, 710 and 180 ppm for propylene carbonate), while the lithium perchlorate water content after vacuum drying was about 0.04 percent (400 ppm). The lithium test piece was a ribbon, $1/2 \times 1/16 \times 1$ ", which had the petrolatum used for protection during storage removed by rinsing with heptane. Results of these studies are shown in Tables III and IV, pages 6 and 7

The detrimental effect of water on the stability of lithium in these solvents is clearly demonstrated in these test series, but the presence of LiClO_4 appears to be beneficial. It had been observed in earlier tests that the presence of LiClO_4 increased the rate of attack. However, the level of water contamination in those tests had not been controlled as rigidly as is done in the present work, and, therefore, the results of those tests could have been seriously affected by water contamination.

Attempts are now being made to produce electrolyte solutions having water contamination levels in the order of 20-50 ppm. It is hoped that a further improvement in lithium stability will be observed in these solutions.

TABLE III

LITHIUM STABILITY IN BUTYROLACTONE AT ROOM TEMPERATURE

Exposure Time	H ₂ O = 2000 ppm No Solute	H ₂ O = 1900 ppm 15g LiClO ₄ /100 ml	H ₂ O = 390 ppm No Solute	H ₂ O = 390 ppm 15g LiClO ₄ /100 ml
10 hours	All Li surfaces black	Slight tarnishing of cut Li only	No visible attack	No visible attack
25 hours	No further changes except for the curling of the Li ribbon	Translucent film covering most of the shiny Li; traces of a gelatinous solid in the liquid	Cut Li only is somewhat blackened	No visible attack
100 hours	Same	Liquid phase full of white gelatinous material	Cut Li completely black; degreased surface blackening	No visible attack
300 hours			2/3 of degreased Li completely black	No attack on any shiny Li, but the amorphous surface initially black is now white and crystalline over 2/3 of its area
500 hours			All Li completely black; Li ribbon curling	No further changes

TABLE IV

LITHIUM STABILITY IN PROPYLENE CARBONATE AT ROOM TEMPERATURE

Exposure Time	H ₂ O = 710 ppm No Solute	H ₂ O = 660 ppm. 15g LiClO ₄ /100 ml	H ₂ O = 180 ppm No Solute	H ₂ O = 190 ppm 15g LiClO ₄ /100 ml
10 hours	Cut surfaces completely black; slight tarnishing of degreased surfaces	No visible attack	Slight tarnishing	No visible attack
25 hours	Translucent film covering most of Li; white gelatinous material in liquid	No visible attack	Cut surfaces completely black	No visible attack
100 hours	White material increased in amount; all shiny Li film covered; Li ribbon curling	Slight tarnishing of cut surfaces only; a trace of white gelatinous material in liquid	Degreased Li blackening	No visible attack
300 hours	No further changes	No further changes	2/3 of degreased Li is black	No visible attack
500 hours			All of Li completely black; traces of white, gelatinous material in liquid; Li ribbon curling	No visible attack

3.1.2. Purification of Propylene Carbonate and Butyrolactone

The method of agitating a solvent in the presence of lithium metal as a means of reducing trace water concentration and decomposing other possible impurities which would otherwise tend to attack the lithium electrode of the cell has been studied extensively on this program. During the past quarter, this procedure was extended to redistillation of the solvents (at the present time, propylene carbonate and butyrolactone) after the lithium powder treatment.

During the lithium treatment phase, the solvents were agitated at room temperature with 1.0g of Li powder/100 grams of solvent in sealed containers. The pressure generated in the containers was relieved periodically, and agitation was continued until no additional pressure build-up was observed. The water content of the solvents was monitored at various intervals by the Karl Fischer analysis, and the results are presented in Table V.

TABLE V
RATE OF WATER REMOVAL IN
PROPYLENE CARBONATE AND BUTYROLACTONE

<u>Butyrolactone</u>		<u>Propylene Carbonate</u>	
<u>Agitation time, hrs.</u>	<u>Water Content, ppm</u>	<u>Agitation time, hrs.</u>	<u>Water Content, ppm</u>
0	470	0	270
6	280	5	140
15	140	17	50*
27	40*	30	23
50	23		

* No further pressure build-up

The excess powdered lithium and the solid reaction products were removed by vacuum filtration through a fine gas washing tube. The insoluble decomposition products had the appearance of a gray, gelatinous mass, and gravitated to the bottom of the liquid phase (the powdered lithium floats).

After removal of the decomposition products and excess powdered lithium, the solvents were vacuum distilled, and the fractions were analyzed.

Results of typical distillation runs for butyrolactone and propylene carbonate are presented in Tables VI and VII. The lithium treatment had caused no observable change in the system; i. e., the behavior of the treated solvents upon distillation was not significantly different from that of the untreated material (NASA CR-54859, pages 8 and 9). The only difference in behavior which could be observed for the Li treated material was a brief period of effervescence at the beginning of the vacuum distillation (possibly caused by the release of dissolved hydrogen).

TABLE VI

VACUUM DISTILLATION OF
LITHIUM-TREATED BUTYROLACTONE

	<u>Boiling Range</u>	<u>Percent Volume</u>	<u>Water, ppm</u>	<u>Color</u>
Undistilled	---	100	23	Off White
Fraction I	21-50°C 8 mm Hg	0	--	Gas
Fraction II	68-72°C 8-10 mm Hg	4.2	1400	White
Fraction III	79-80°C 10 mm Hg	89.9	23	White
Fraction IV	---	5.9	---	Brown liquid Gray solids

TABLE VII

VACUUM DISTILLATION OF
LITHIUM-TREATED PROPYLENE CARBONATE

	<u>Boiling Range</u>	<u>Percent Volume</u>	<u>Water, ppm</u>	<u>Color</u>
Undistilled	---	100	23	Off White
Fraction I	23-50°C 10-15 mm Hg	0	--	Gas
Fraction II	96-108°C 6-10 mm Hg	7.4	230	White
Fraction III	106-108°C 10 mm Hg	89.3	23	White
Fraction IV	---	3.3	--	Brown liquid Gray solids

The brown, oily liquid residue is characteristic to BL and PC, and occurs also during the distillation of untreated solvents. The gray precipitate, however, indicates production of soluble lithium compounds during the agitation period.

The high water content in Fraction II of the butyrolactone distillation was consistently obtained in three separate distillation runs. Assuming all of the water of the system to be in this fraction, 60 ppm in the original material is indicated rather than the less than 23 ppm determined after lithium treatment. Unless a material reactive to the Karl Fischer reagent has been generated by the lithium treatment, this would indicate contamination of the material during or just before distillation. It was clearly shown, however, that the first 5-10 percent of distillate is very high in water content, and, therefore, a substantial reduction in water content can be achieved by rejecting this portion of the distillate.

The solvents which are being used in this program are very pure and are not readily separable into fractions for which differences in physical properties such as boiling point or index of refraction can be readily detected. Consequently, the standard procedure for preparing butyrolactone and propylene carbonate for cell tests consists of agitation of the solvent with a small amount of lithium powder (ca. 5-10 g/L) and subsequent vacuum distillation rejecting the initial and final 10 percent of the distillate. By this method, solvents having water content below the sensitivity of the Karl Fischer analysis (20 ppm) can be prepared.

3.1.3. Solubility of CuF_2 in Propylene Carbonate and Butyrolactone

A certain degree of solubility of CuF_2 in the electrolyte solutions has been evident from a number of previous tests, and from the green-blue appearance of cell electrolytes after discharge. It has also been apparent that the extent of this solubility is dependent on the amount of water contamination of the system, i.e., the solubility is increased with increasing water content. During the past quarter, a quantitative study of this phenomenon was initiated.

A series of 60 ml glass serum bottles were filled with 40 ml of LiClO_4 -PC or LiClO_4 -BL solutions of known water content. To these, 1.0g of CuF_2 was added, the water content of which had been determined by X-ray analysis. Varying amounts of water were then added by a micro syringe to the samples to give calculated water concentrations over the range of 250-8600 ppm. The samples thus prepared were placed on a laboratory shaker and, after an agitation period followed by a rest period to allow the solid CuF_2 to settle out, the solutions were analyzed for copper ion by the thio-sulfate titration.

The Karl Fischer analysis was also performed on the extract, but no significance can as yet be assigned to the results obtained. An exact correction for the presence of copper ion cannot be made; also, a substance reactive to the Karl Fischer reagent is produced during the agitation period as indicated by Karl Fischer titers which are much higher than would result from titrating all of the water introduced into the system at the beginning of the test.

From Tables VIII and IX, pages 12 and 13, the solubility of CuF_2 in both the propylene carbonate and the butyrolactone solutions can be seen to increase with increasing water content of the original solution. This was also evidenced by a more intense blue-green coloration of the samples having the higher water content. In the samples which had less than 60 μmols of water/ml, little difference in copper solubility was obtained between butyrolactone and propylene carbonate solvents. With the higher water contents of 155 and 420 $\mu\text{mols}/\text{ml}$, concentration of Cu^{++} after 600 hours of agitation was about 50 percent higher in butyrolactone than in propylene carbonate. None of the systems appeared to have reached an equilibrium Cu^{++} concentration, after 600 hours of agitation.

From Tables VIII and IX, it can be seen that the mole ratio of the initial total water content of the samples to the copper ion in solution is tending toward a constant value—i. e., the amount of copper which exists in the solution appears to be a function of the amount of water in the system. This observation further demonstrates the need for a very low water contamination level in CuF_2 -Li cells with shelf-life requirements. It is intended to supplement the work described in the present report with solubility tests employing materials which give a solution water content in the range of 1-5 $\mu\text{mols}/\text{ml}$, which, at the present, approximates the lower practical limit of water control in these systems.

TABLE VIII
SOLUBILITY OF CuF_2 IN PC/LiClO_4 ELECTROLYTE

Materials and Weights:

1.00g CuF_2	= (3,300 \pm 500) $\mu\text{g H}_2\text{O}$	Ozark Mahoning; H_2O by X-ray
4.00g LiClO_4	= (10,800 \pm 400) $\mu\text{g H}_2\text{O}$	Foot Mineral; H_2O by KFA
48.2 g PC	= (480 \pm 480) $\mu\text{g H}_2\text{O}$	MC & B; Li dried & distilled H_2O by KFA
52.2g of solu- tion in each sample	(14,600 \pm 1400) $\mu\text{g H}_2\text{O}$ in each sample	

Solution Density = 1.25g/ml

	<u>Agitation Time, hrs.</u>	<u>(Cu^{++}) ($\mu\text{mols/ml}$)</u>	<u>(H_2O)/(Cu^{++}) (mole ratio)</u>	<u>Color of Solution</u>
Sample I	25	5.0 \pm 0.1	4.0 \pm 0.4	Off White
$\text{H}_2\text{O} = 20 \pm 2$	125	5.8 \pm 0.1	3.5 \pm 0.4	Off White
$\mu\text{mols/ml}$	290	9.8 \pm 0.1	2.0 \pm 0.2	Olive Yellow
	650	14.1 \pm 0.2	1.4 \pm 0.1	
Sample II	25	5.5 \pm 0.1	5.1 \pm 0.5	Off White
$\text{H}_2\text{O} = 28 \pm 2$	125	7.0 \pm 0.1	4.0 \pm 0.4	Off White
$\mu\text{mols/ml}$	290	11.5 \pm 0.2	2.4 \pm 0.2	Olive Yellow
	650	14.2 \pm 0.2	2.0 \pm 0.2	
Sample III	25	6.0 \pm 0.1	9.3 \pm 0.5	Off White
$\text{H}_2\text{O} = 56 \pm 3$	125	11.0 \pm 0.2	5.1 \pm 0.3	Off White
$\mu\text{mols/ml}$	290	15.7 \pm 0.2	3.6 \pm 0.2	Olive Yellow
	650	20.0 \pm 0.2	2.8 \pm 0.2	
Sample IV	25	18.0 \pm 0.2	8.6 \pm 0.1	Pale Green
$\text{H}_2\text{O} = 154 \pm 3$	125	32.5 \pm 0.3	4.7 \pm 0.1	Pale Green
$\mu\text{mols/ml}$	290	40.0 \pm 0.4	3.9 \pm 0.1	Olive Green
	650	68 \pm 2	2.3 \pm 0.1	
Sample V	25	101 \pm 2	4.2 \pm 0.2	Blue
$\text{H}_2\text{O} = 420 \pm 4$	125	113 \pm 1	3.7 \pm 0.1	Blue Green
$\mu\text{mols/ml}$	290	118 \pm 2	3.6 \pm 0.1	Green
	650	145 \pm 3	2.9 \pm 0.1	

TABLE IX
SOLUBILITY OF CuF_2 IN BL/LiClO_4 ELECTROLYTE

Materials and Weights:

1.00g CuF_2	= (3,300 \pm 500) $\mu\text{g H}_2\text{O}$	Ozark Mahoning; H_2O by X-ray
4.00g LiClO_4	= (10,800 \pm 400) $\mu\text{g H}_2\text{O}$	Foote Mineral; H_2O by KFA
45.0 g BL	= (450 \pm 450) $\mu\text{g H}_2\text{O}$	MC & B; Li dried & distilled
49.0 g of solu-	(14,600 \pm 1,400) $\mu\text{g H}_2\text{O}$	H_2O by KFA
tion in each	in each	
sample	sample	

Solution Density = 1.18 g/ml

	<u>Agitation Time, hrs.</u>	<u>(Cu^{++}) ($\mu\text{mols/ml}$)</u>	<u>(H_2O)/(Cu^{++}) (mole ratio)</u>	<u>Color of Solution</u>
Sample I	25	3.0 \pm 0.1	6.7 \pm 0.7	Off White
$\text{H}_2\text{O} = 20 \pm 2$	125	4.0 \pm 0.1	5.0 \pm 0.5	Off White
$\mu\text{mols/ml}$	290	8.0 \pm 0.1	2.5 \pm 0.3	Yellow Green
	650	11.8 \pm 0.2	1.7 \pm 0.2	
Sample II	25	4.0 \pm 0.1	7.0 \pm 0.7	Off White
$\text{H}_2\text{O} = 28 \pm 2$	125	4.5 \pm 0.1	6.2 \pm 0.6	Off White
$\mu\text{mols/ml}$	290	10.0 \pm 0.1	2.8 \pm 0.3	Yellow Green
	650	14.0 \pm 0.2	2.0 \pm 0.2	
Sample III	25	5.0 \pm 0.1	11.2 \pm 0.5	Very Pale Yellow
$\text{H}_2\text{O} = 56 \pm 3$	125	8.8 \pm 0.1	6.4 \pm 0.3	Pale Green
$\mu\text{mols/ml}$	290	17.9 \pm 0.2	3.1 \pm 0.2	Olive Green
	650	22.7 \pm 0.2	2.5 \pm 0.2	
Sample IV	25	18.0 \pm 0.2	8.6 \pm 0.1	Pale Green
$\text{H}_2\text{O} = 154 \pm 3$	125	31.0 \pm 0.3	5.0 \pm 0.1	Green
$\mu\text{mols/ml}$	290	53.0 \pm 0.4	2.9 \pm 0.1	Green
	650	90 \pm 3	1.7 \pm 0.1	
Sample V	25	113 \pm 2	3.7 \pm 0.1	Blue
$\text{H}_2\text{O} = 422 \pm 4$	125	125 \pm 1	3.4 \pm 0.1	Blue Green
$\mu\text{mols/ml}$	290	162 \pm 3	2.6 \pm 0.1	Blue Green
	650	226 \pm 4	1.9 \pm 0.1	

3.2. POSITIVE ELECTRODE STUDIES

3.2.1. Pasted CuF_2 Electrode Tests

In order to achieve the desired performance of 50 watt hours/lb in the discharge rate range of 1-10 hours, a thin-plate CuF_2 -Li cell will have to be developed. Since little difficulty is visualized in producing thin lithium electrodes (0.015" sheet is available commercially), the present work is proceeding for the development of a thin CuF_2 electrode. The present electrodes are prepared by pasting a mixture of CuF_2 , graphite, and cellulose acetate wetted with 90 percent ethyl acetate - 10 percent ethyl alcohol solution onto expanded silver support. The resulting electrodes have a thickness of about 0.025" and an area of 1.5" x 1.5", or about 30 cm^2 when assembled with two negative electrodes in three-plate cells. To insure positive limitation, the lithium electrodes have a combined theoretical capacity of about 4 ampere hours compared to 0.5 - 1.0 ampere hour in the CuF_2 electrode. The "standard" separation in these cells is 0.02" thick glass fiber mat, and 50g LiClO_4 /100 ml methyl formate is employed as the electrolyte. The cells have polyethylene outside cases, and are placed between blocks for lateral support. The cell components, an assembled cell, and a number of cells assembled for discharge are pictured in Figures 1 to 3, pages 16 to 18.

During the past quarter, discharge tests with pasted CuF_2 electrodes were performed at current densities of 5-15 mA/cm^2 . Test temperature was generally -15°C in order to minimize solvent loss during discharge.

To a final cell voltage of 2.0 volts, electrochemical efficiencies in the range of 50-70 percent were obtained for the CuF_2 electrodes. The initial and average discharge potential of the cells increased with increasing graphite content of the paste. As expected, increasing the current density from 10 to 15 mA/cm^2 decreased the discharge potential; however, the electrochemical efficiency was not significantly affected over the current density range of 5.0 to 15.0 mA/cm^2 . Increasing the thickness of the electrodes from 0.025" to 0.050" decreased both the discharge potential and the electrochemical efficiency. Lithium perchlorate added to the paste in amounts of 3.0 to 12.0 g/100g of CuF_2 also showed no noticeable effect on the discharge properties of the electrodes.

It may be seen from the tabulation of cell construction data that the theoretical capacity of the 1.5 x 1.5 x 0.025 inch CuF_2 electrodes increases as the graphite content in the paste is decreased, while the electrolyte volume required for the cell remains essentially the same. Therefore, the theoretical energy density of a cell will change with CuF_2 electrode composition. This has to be considered, in addition to the efficiency of CuF_2 utilization, in the evaluation of the mix composition. At the present, one gram of cellulose acetate and 10-15 grams of graphite per 100 grams of CuF_2 represents an optimum composition range for the discharge rates employed.

Further development in this area will include studies of other additives and/or binders in order to improve the voltage regulation and efficiency of the CuF_2 electrodes. At the conclusion of single electrode evaluation, cells having larger capacity (5 AH) will be constructed and tested.

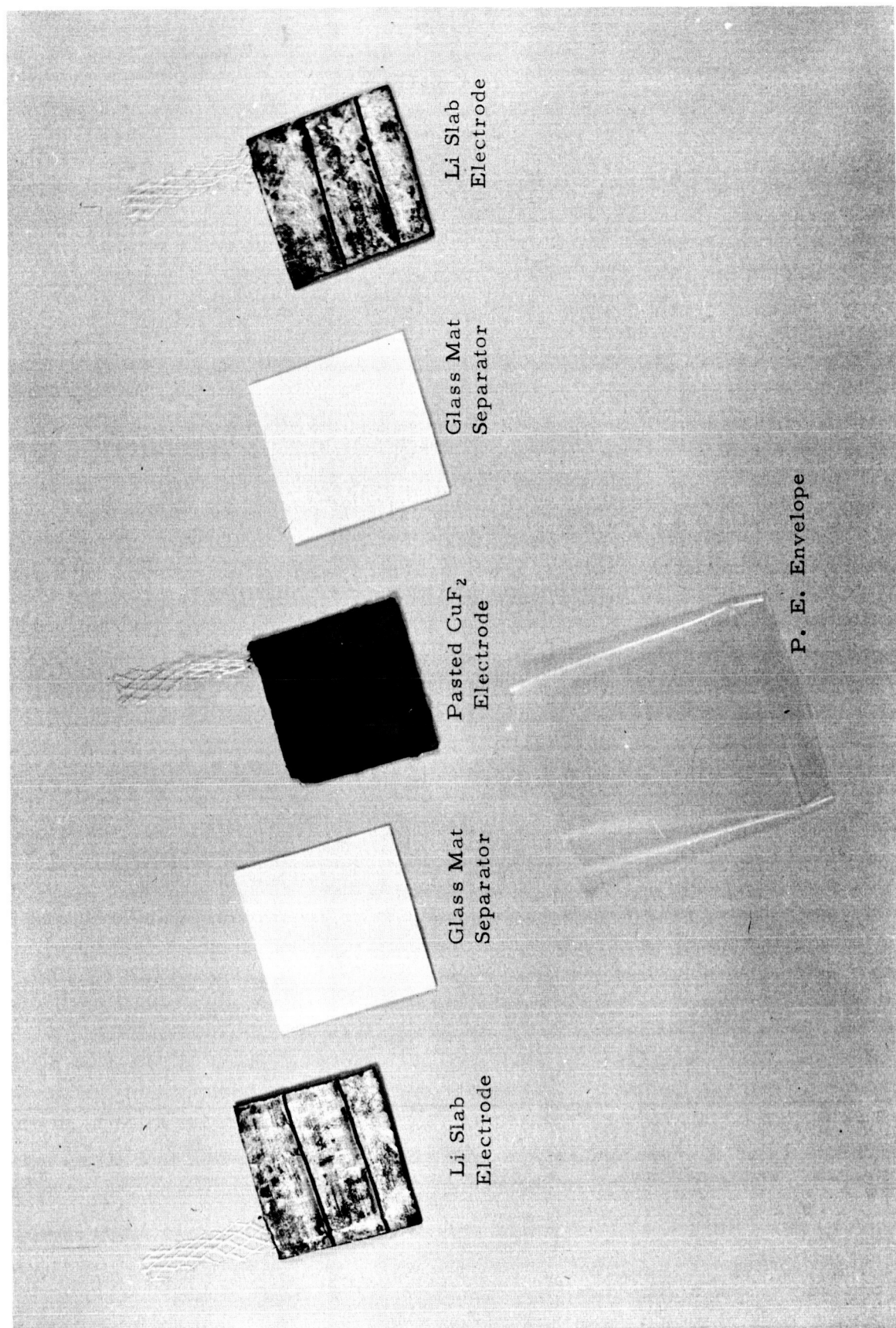
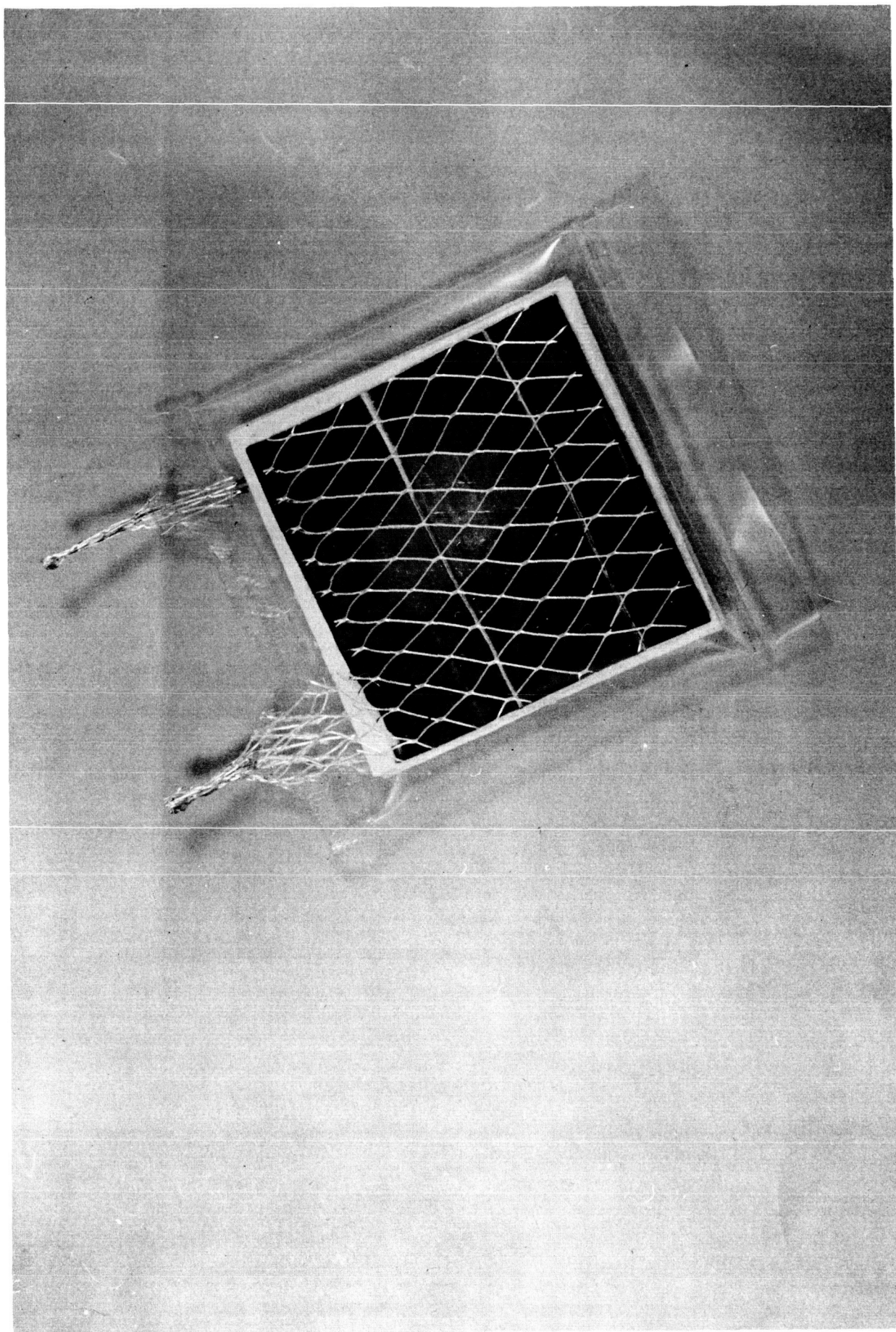
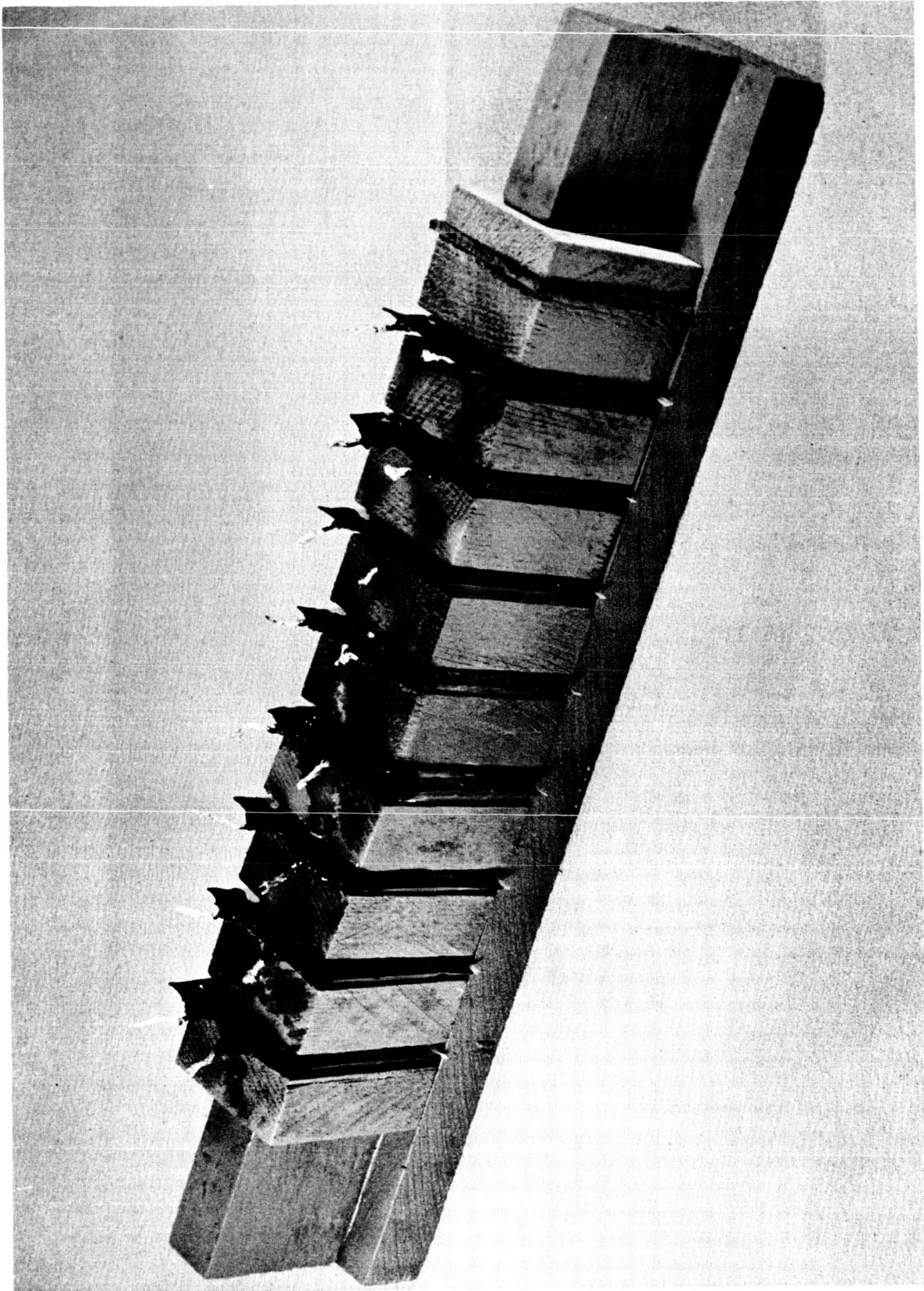
PASTED CuF_2 ELECTRODE TEST CELL COMPONENTS

FIGURE 1



PASTED CuF_2 ELECTRODE TEST CELL

FIGURE 2



CELL DISCHARGE RACK

FIGURE 3

TABLE X
CELL CONSTRUCTION AND

Cell No.	Gms. additive/100g of CuF_2		Dry paste Wt., gms.	CuF_2 Capacity, AH	Electrolyte Vol., cc	Open Circuit Potential
	Graphite	Cellulose Acetate				
1-1	20	2	1.27	0.55	3.0	3.53
1-2	20	2	1.13	0.49	3.0	3.55
1-3	20	2	1.40	0.61	2.7	3.52/3.35
1-4	20	2	1.30	0.56	2.9	3.53
1-5	20	2	1.42	0.62	3.4	3.53
1-6	20	2	1.44	0.63	3.3	3.53
1-7	20	2	2.20	0.95	3.5	3.53
1-8	20	2	2.10	0.91	2.1	3.53
1-9	20	2	2.26	0.98	3.7	3.51
1-10	20	2	2.12	0.92	4.5	3.51
2-1	10	2	1.62	0.76	2.8	3.37
2-2	10	2	1.77	0.84	2.8	3.49
2-3	10	2	1.54	0.73	3.0	3.49
2-4	10	2	1.60	0.76	2.8	3.48
2-5	10	2	1.63	0.77	2.5	3.54
2-6	10	2	1.57	0.74	2.7	3.52
2-7	10	2	1.47	0.69	2.5	3.52
2-8	10	2	1.53	0.72	2.6	3.57
3-1	5	2	1.32	0.65	2.5	3.51
3-2	5	2	1.47	0.73	2.5	3.51
3-3	5	2	1.40	0.69	2.5	3.51
3-4	5	2	1.70	0.84	Leak	3.51
3-5	5	2	1.69	0.84	2.5	3.51
3-6	5	2	1.90	0.94	3.0	3.51
4-1	2.5	2	1.59	0.81	3.0	3.47
4-2	2.5	2	1.41	0.72	3.0	3.47
4-3	2.5	2	1.50	0.76	3.0	3.47
4-4	2.5	2	1.52	0.77	3.0	3.47
4-5	2.5	2	1.69	0.86	4.0	3.37
4-6	2.5	2	1.59	0.81	3.6	3.21

DISCHARGE DATA

Discharge Potential		Capacity to 2.0VF, AH	Electrochemical Efficiency, %	Remarks
Initial	Average			
2.91	2.67	0.37	67	
2.88	2.67	0.34	69	
2.86	2.52	0.28	40	Apparent internal short
2.49	2.24	0.25	45	MPR Separation
2.74	2.27	0.24	38	MPR Separation
2.46	2.24	0.32	50	MPR Separation
2.82	2.56	0.64	67	
2.78	2.56	0.55	60	
2.39	2.25	0.33	33	MPR separation, .040" positive
2.00	2.00	---	--	MPR separation, .040" positive
2.89	2.63	0.44	58	
2.76	2.67	0.57	68	
2.85	2.72	0.47	65	
2.81	2.65	0.50	66	
2.81	2.73	0.51	66	} Room temperature sealed Room temperature sealed MPR separation-room temp. MPR separation-room temp.
2.82	2.65	0.38	51	
2.58	2.50	0.38	55	
2.53	2.66	0.54	75	
2.76	2.70	0.43	66	
2.76	2.72	0.44	59	
2.58	2.66	0.43	62	
2.69	2.48	0.28	33	Leak upon activation
2.58	2.50	0.31	37	
2.58	2.59	0.58	62	
2.72	2.58	0.49	60	
2.67	2.59	0.39	54	
2.73	2.65	0.50	66	
2.69	2.60	0.45	58	
2.70	2.45	0.15	17	2.35M Electrolyte
2.61	2.46	0.20	25	2.35M Electrolyte

TABLE X
CELL CONSTRUCTION AND

Cell No.	Grams additive/ 100g of CuF_2		Dry paste Wt., gms.	CuF_2 Capacity, AH	Electrolyte Volume, cc	Open Circuit Potential
	Graphite	Cellulose Acetate				
5-1	20	1	1.20	0.52	3.0	3.58
5-2	20	1	1.36	0.59	3.0	3.56
5-3	20	1	1.24	0.54	3.0	3.57
5-4	20	1	1.29	0.56	3.0	3.57
6-1	10	1	1.36	0.65	3.0	3.52
6-2	10	1	1.39	0.66	3.0	3.56
6-3	10	1	1.47	0.70	3.0	3.58
6-4	10	1	1.62	0.77	3.0	3.56
7-1	5	1	1.64	0.82	3.0	3.50
7-2	5	1	1.40	0.70	3.0	3.50
7-3	5	1	1.86	0.93	3.0	3.52
7-4	5	1	1.58	0.79	3.0	3.51
8-1	2.5	1	1.80	0.92	3.0	3.48
8-2	2.5	1	1.88	0.96	3.0	3.51
8-3	2.5	1	1.88	0.96	3.0	3.49
8-4	2.5	1	1.88	0.96	3.0	3.49
9-1	20	0.5	1.29	0.57	3.0	3.50
9-2	20	0.5	1.32	0.58	3.0	3.53
9-3	20	0.5	1.23	0.54	3.0	3.54
9-4	20	0.5	1.31	0.58	3.0	3.54
10-1	10	0.5	1.51	0.73	3.0	3.48
10-2	10	0.5	1.61	0.77	3.0	3.48
10-3	10	0.5	1.66	0.80	3.0	3.53
10-4	10	0.5	1.73	0.83	3.0	3.49
11-1	5	0.5	1.65	0.83	3.0	3.42
11-2	5	0.5	2.01	1.01	3.0	3.42
11-3	5	0.5	1.96	0.98	3.0	3.48
11-4	5	0.5	1.70	0.85	3.0	3.48

CONTINUED
DISCHARGE DATA

Discharge Potential		Capacity to 2.0VF, AH	Electrochemical Efficiency, %	Remarks
Initial	Average			
2.84	2.70	0.32	61	
2.81	2.64	0.38	64	
2.75	2.64	0.34	63	
2.78	2.69	0.37	64	
2.73	2.70	0.38	58	
2.77	2.66	0.38	58	
2.74	2.68	0.41	59	
2.65	2.63	0.46	60	
2.64	2.68	0.41	50	
2.58	2.66	0.38	54	
2.57	2.67	0.47	51	
2.60	2.68	0.42	53	
2.32	2.63	0.50	54	
2.29	2.64	0.48	50	
2.25	2.57	0.50	52	
2.35	2.62	0.50	52	
2.94	2.68	0.34	60	
2.94	2.70	0.36	62	
2.90	2.61	0.32	59	
2.82	2.59	0.23	40	Very noisy discharge
2.64	2.68	0.43	59	
2.58	2.67	0.45	58	
2.61	2.58	0.43	54	
2.61	2.64	0.47	57	
2.41	2.65	0.50	60	
2.49	2.67	0.60	60	
2.43	2.63	0.52	53	
2.43	2.62	0.49	58	

TABLE X
CELL CONSTRUCTION AND

Cell No.	grams additive/100g of CuF_2		Dry paste wt., gms.	CuF_2 capacity, AH	Electrolyte Volume, cc	Open Circuit Potential
	Graphite	Cellulose Acetate				
12-1	2.5	0.5	1.74	0.90	3.0	3.37
12-2	2.5	0.5	1.50	0.78	3.0	3.36
12-3	2.5	0.5	1.84	0.95	3.0	3.42
12-4	2.5	0.5	1.82	0.94	3.0	3.36
13-1	10	1	1.63*	0.76	3.0	3.50
13-2	10	1	1.78*	0.83	3.0	3.50
13-3	10	1	1.53*	0.71	3.0	3.50
13-4	10	1	1.45*	0.67	3.0	3.50
13-5	10	1	1.51*	0.70	3.0	3.50
13-6	10	1	1.85*	0.86	3.0	3.50
13-7	10	1	3.63*	1.69	3.8	3.50
13-8	10	1	3.65*	1.70	3.8	3.50
14-1	10	1	1.66*	0.76	3.0	3.45
14-2	10	1	1.93*	0.88	3.0	3.45
14-3	10	1	1.65*	0.75	3.0	3.45
14-4	10	1	1.89*	0.86	3.0	3.45
14-5	10	1	1.78*	0.81	3.0	3.47
14-6	10	1	1.93*	0.88	3.0	3.49
14-7	10	1	1.92*	0.87	3.0	3.56
14-8	10	1	1.95*	0.89	3.0	3.49
15-1	10	1	1.88*	0.83	3.0	3.51
15-2	10	1	1.73*	0.77	3.0	3.51
15-3	10	1	1.94*	0.86	3.0	3.51
15-4	10	1	1.79*	0.79	3.0	3.51
15-5	10	1	1.86*	0.83	3.0	3.56
15-6	10	1	1.91*	0.85	3.0	3.56
15-7	10	1	1.76*	0.78	3.0	3.56
15-8	10	1	1.87*	0.83	3.0	3.56
16-1	10	1	1.60*	0.69	3.0	3.50
16-2	10	1	1.84*	0.80	3.0	3.50
16-3	10	1	1.71*	0.74	3.0	3.50
16-4	10	1	1.58*	0.69	3.0	3.50

*Includes weight of LiClO_4

CONTINUED
DISCHARGE DATA

Discharge Potential		Capacity to 2.0VF, AH	Electrochemical Efficiency, %		Remarks
Initial	Average				
1.88	2.62	0.45	50		
1.88	2.54	0.38	47		
1.95	2.58	0.45	47		
1.93	2.55	0.40	43		
2.79	2.72	0.40	53	}	3g LiClO ₄ /100g CuF ₂
2.78	2.76	0.52	63		
2.55	2.69	0.48	68		
2.61	2.74	0.46	69		
2.67	2.71	0.49	70		
2.67	2.69	0.56	65		
2.13	2.53	0.72	43		
1.99	2.54	0.65	39		
2.88	2.70	0.38	50	}	6g LiClO ₄ /100g CuF ₂
2.82	2.52	0.44	50		
2.87	2.75	0.41	55		
2.87	2.72	0.47	55		
3.06	2.73	0.40	49		
3.08	2.78	0.48	55		
2.64	2.54	0.51	59		
2.58	2.62	0.51	57		
2.88	0.69	0.45	54	}	9g LiClO ₄ /100g CuF ₂
2.90	0.69	0.44	57		
2.89	2.71	0.50	58		
2.89	2.72	0.46	58		
3.14	2.80	0.44	53		
3.14	2.73	0.47	55		
2.67	2.62	0.50	64		
2.63	2.57	0.48	58		
2.73	2.62	0.38	55	}	12g LiClO ₄ /100g CuF ₂
2.70	2.62	0.46	57		
2.72	2.64	0.41	55		
2.70	2.58	0.37	54		

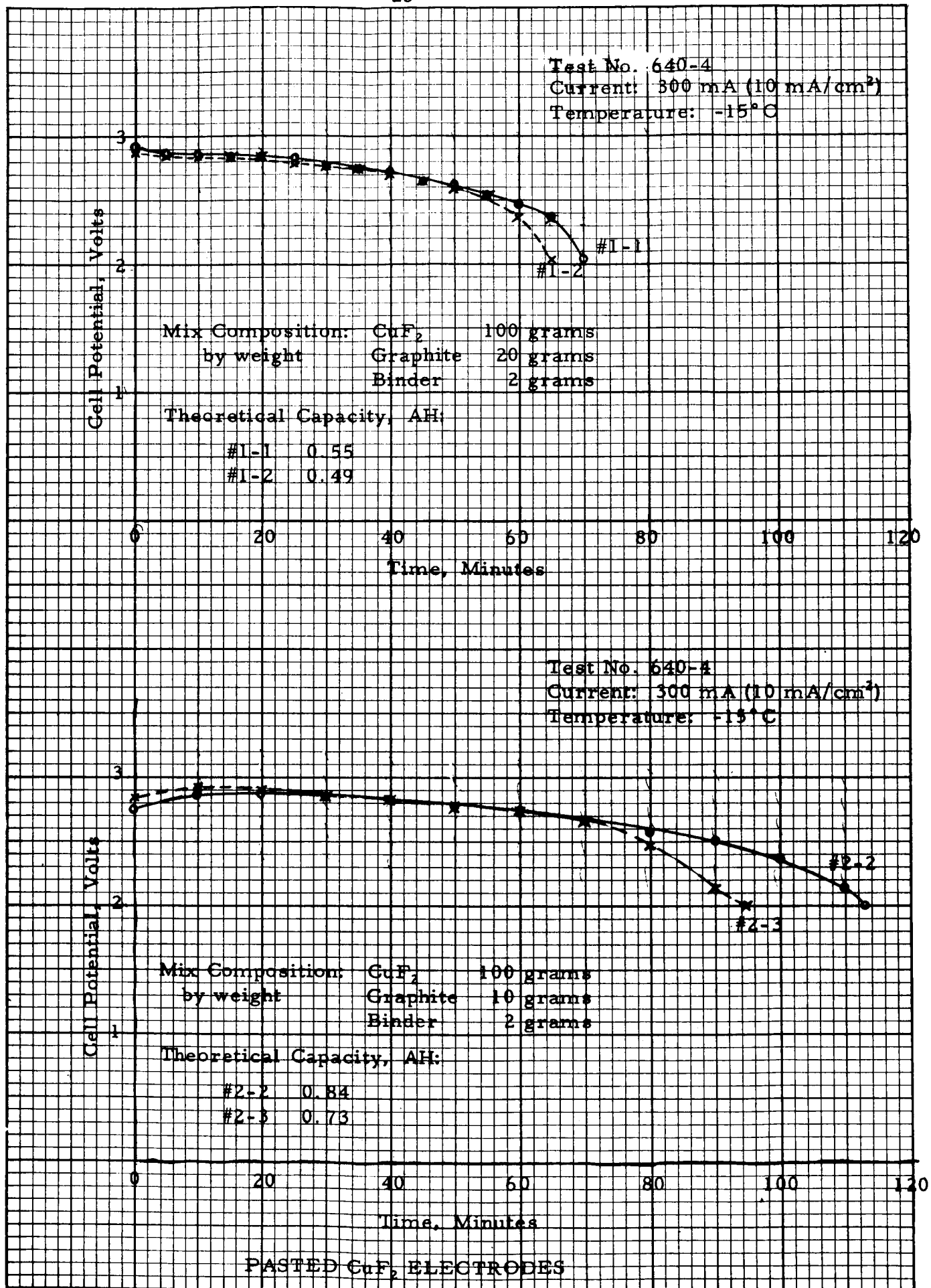


FIGURE 4

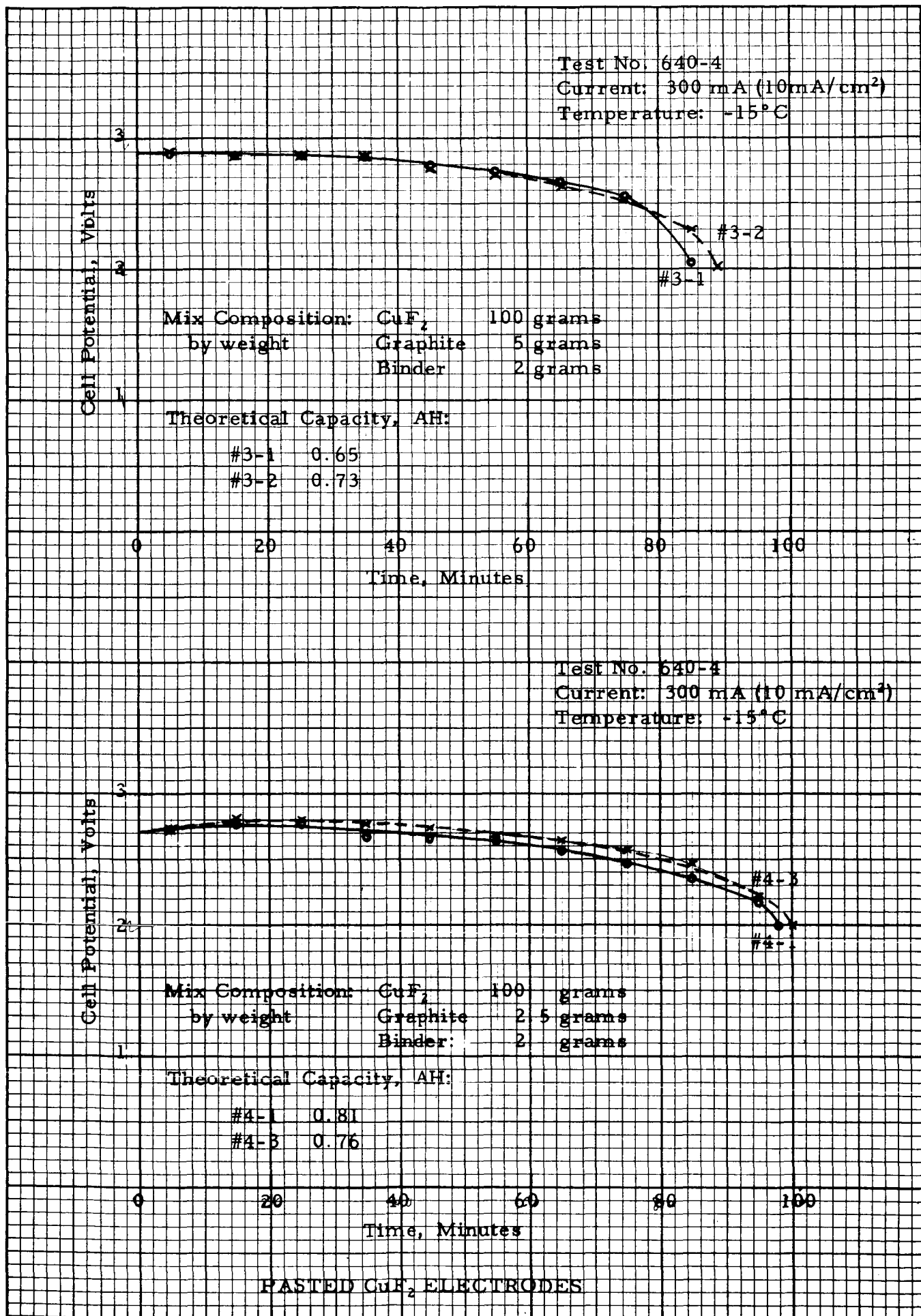
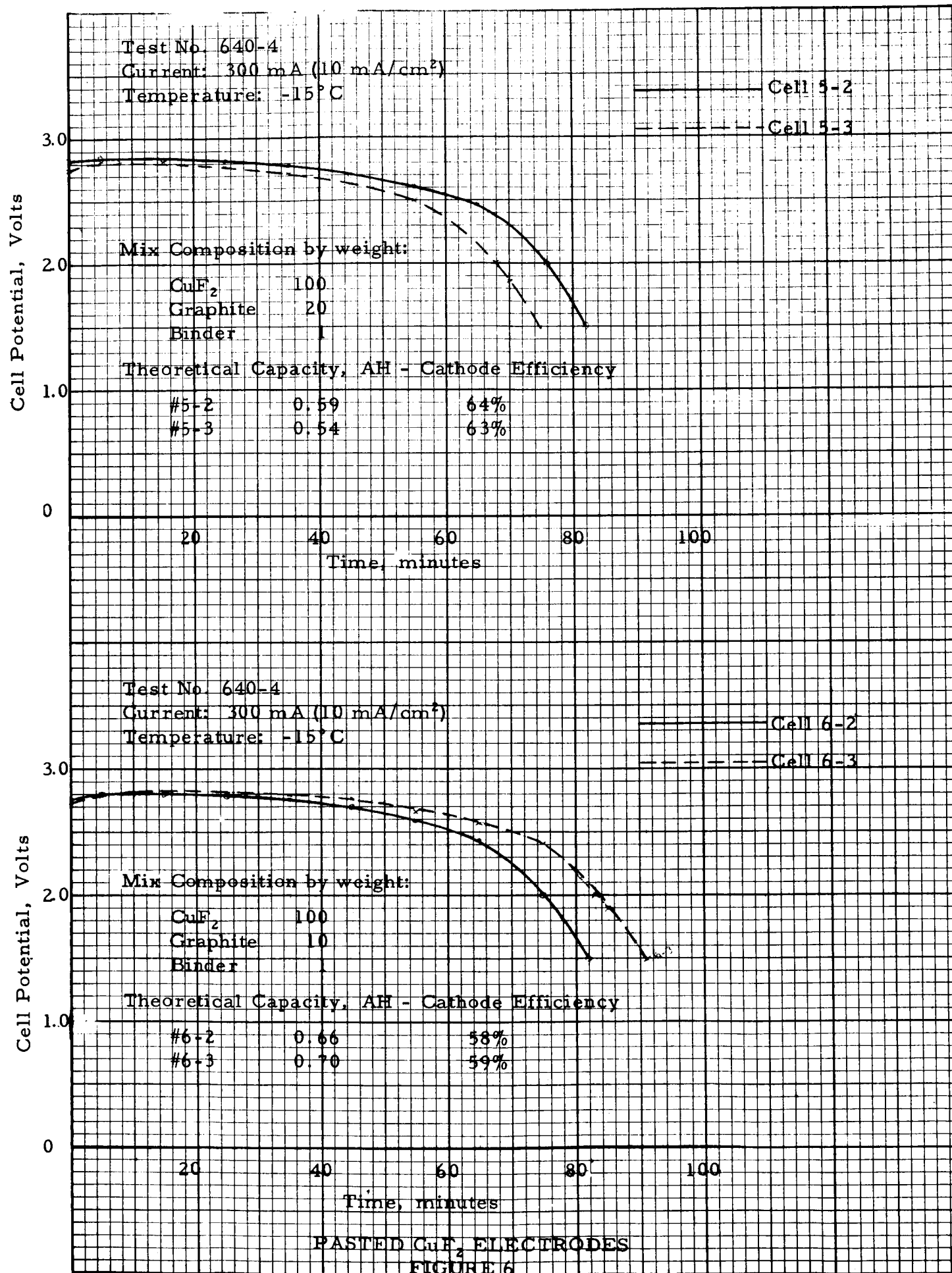
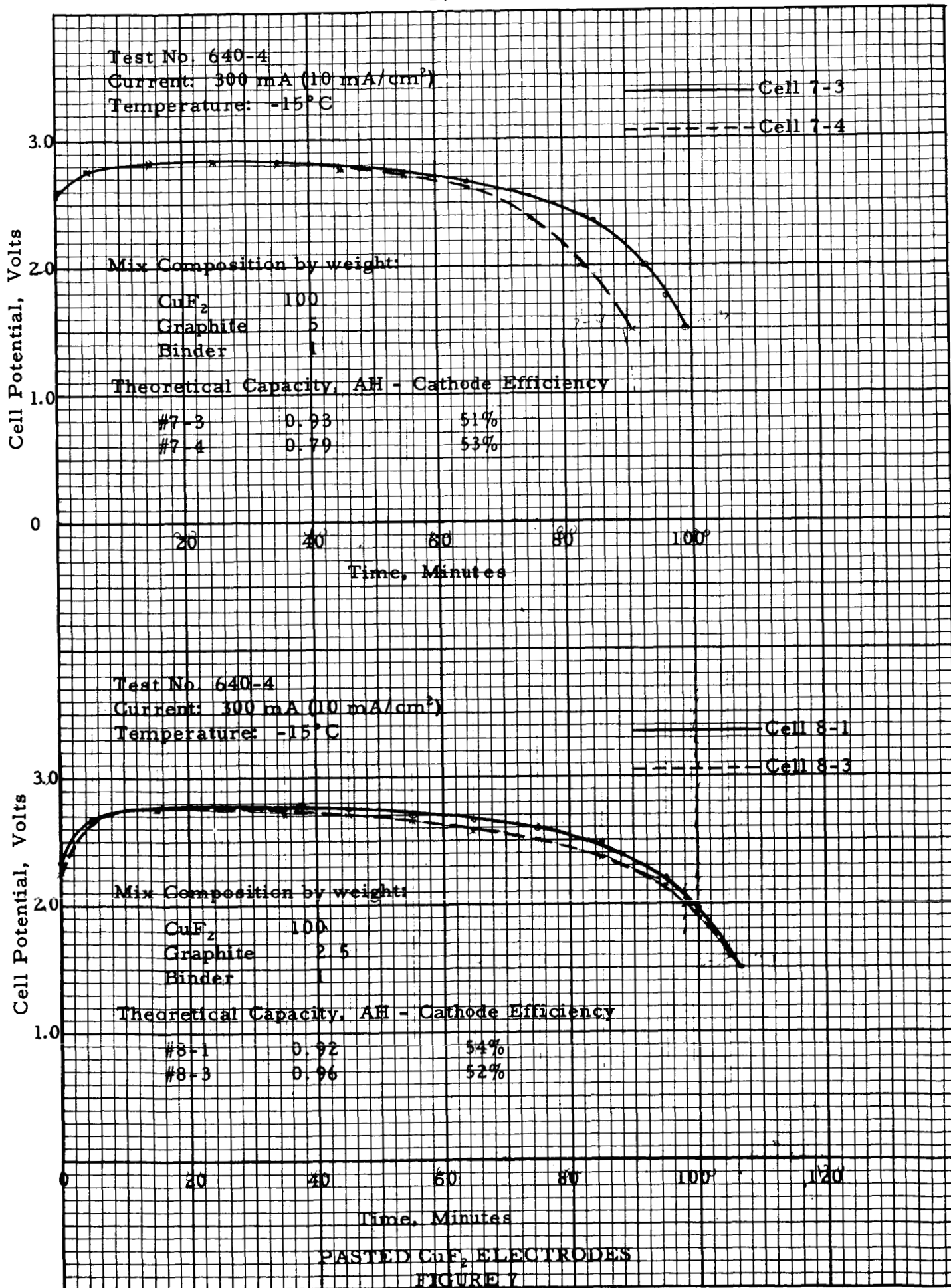
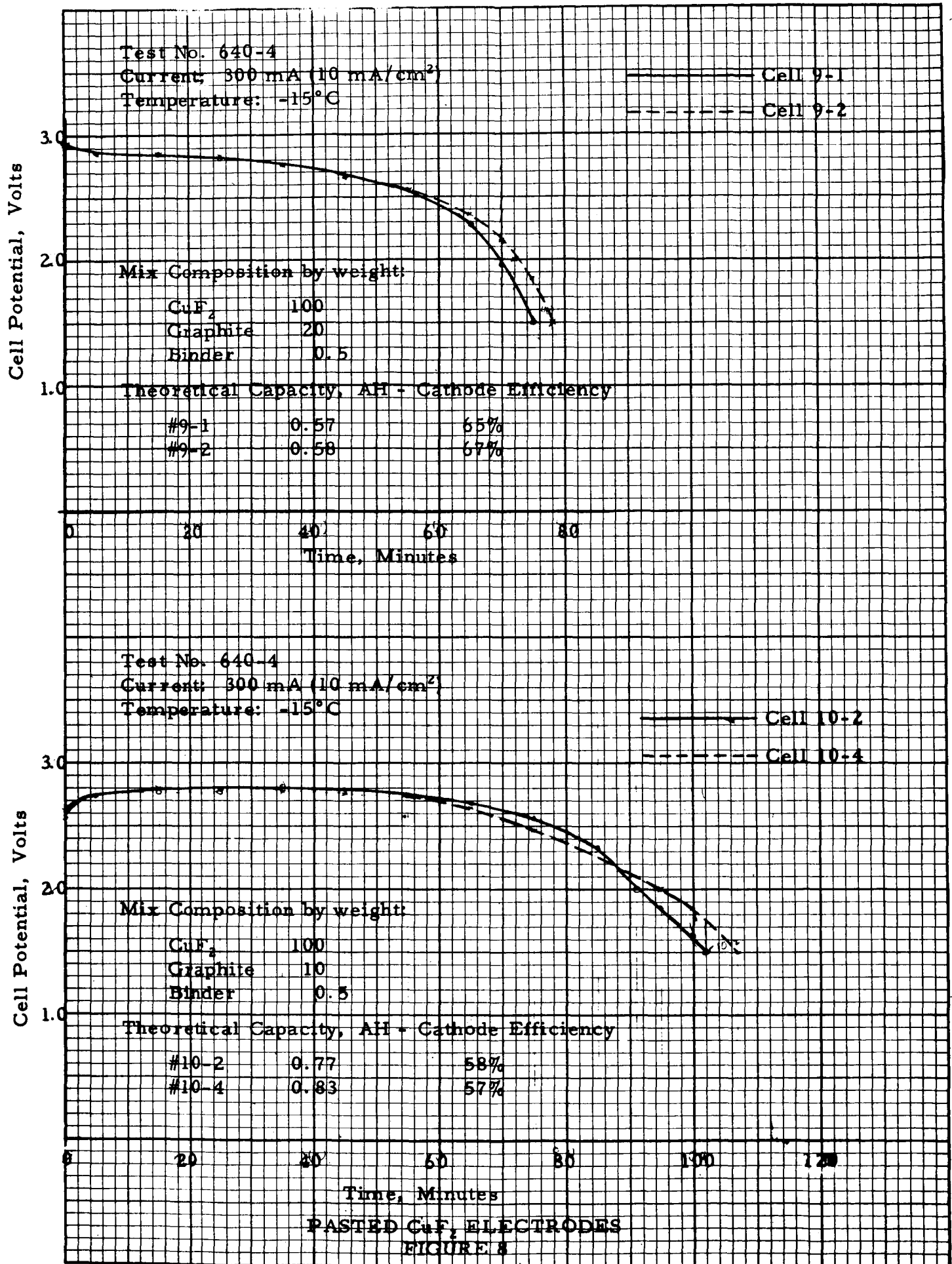
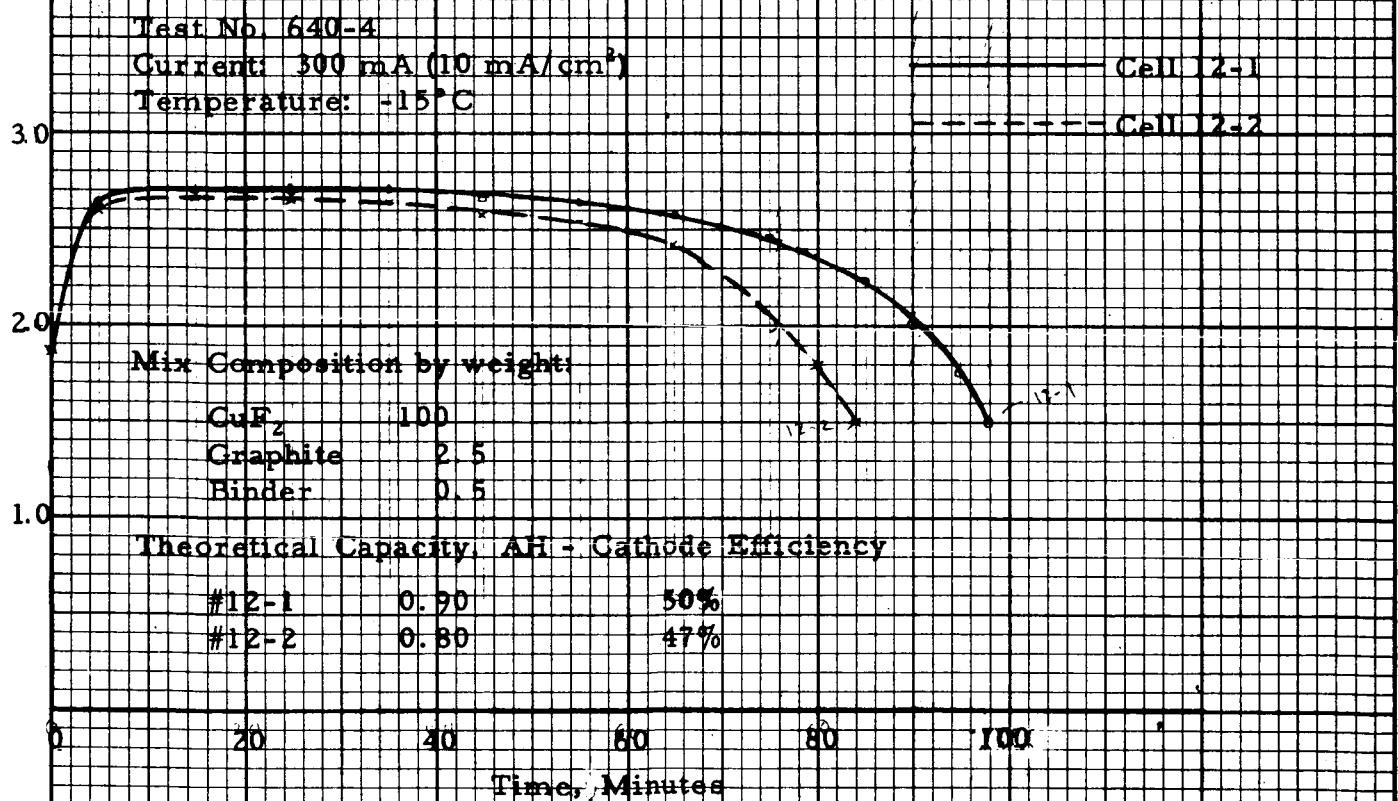
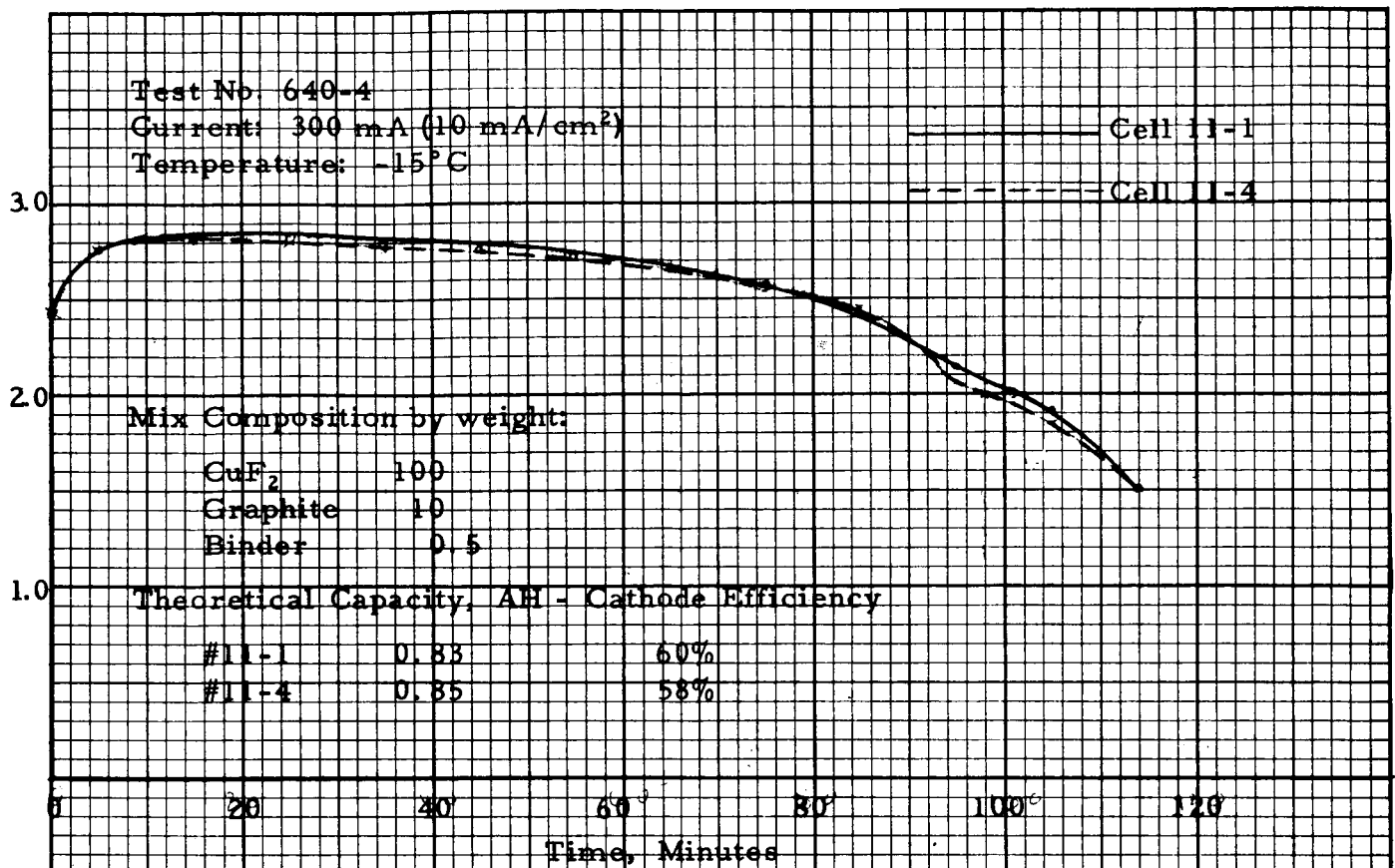


FIGURE 5









PASTED CuF₂ ELECTRODES
FIGURE 9

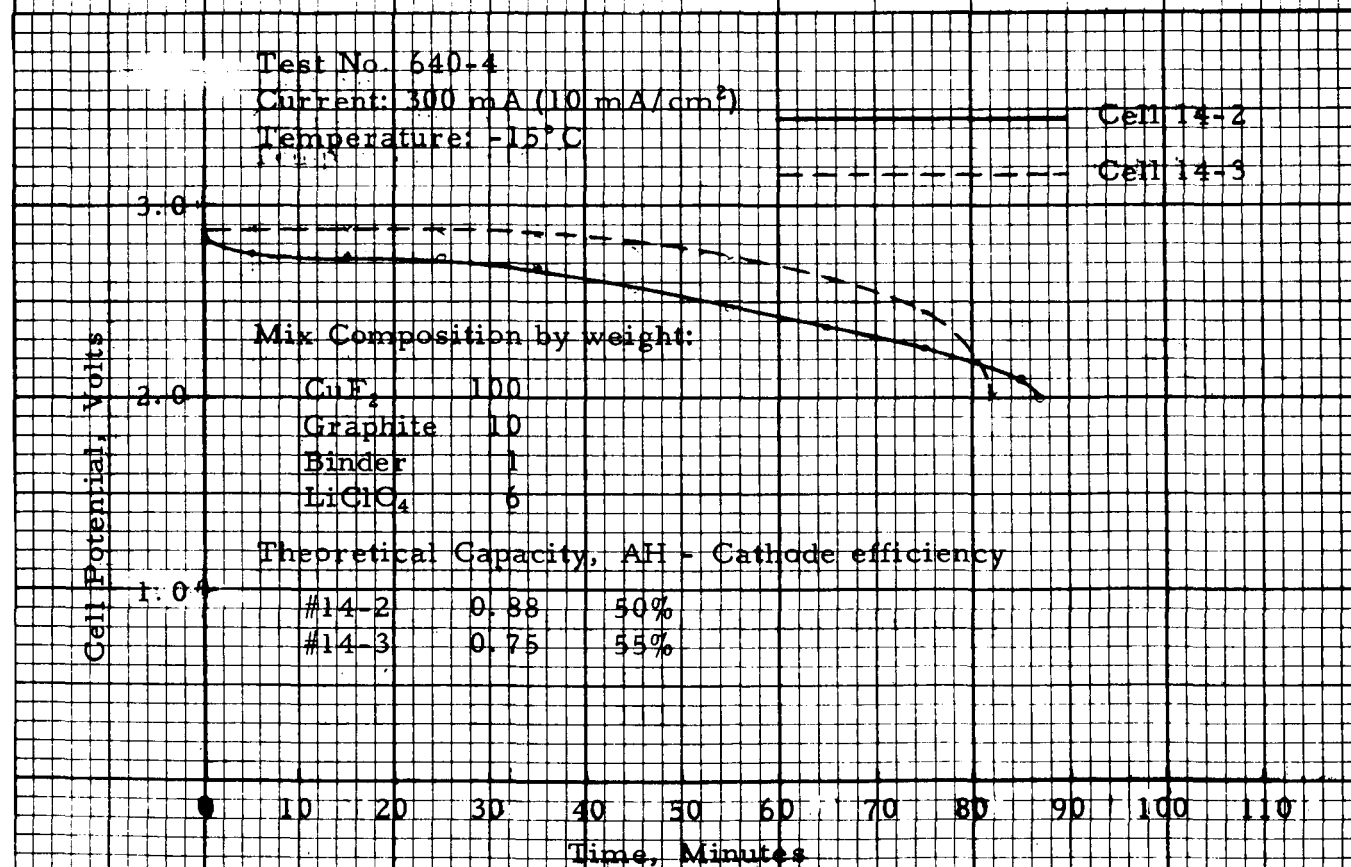
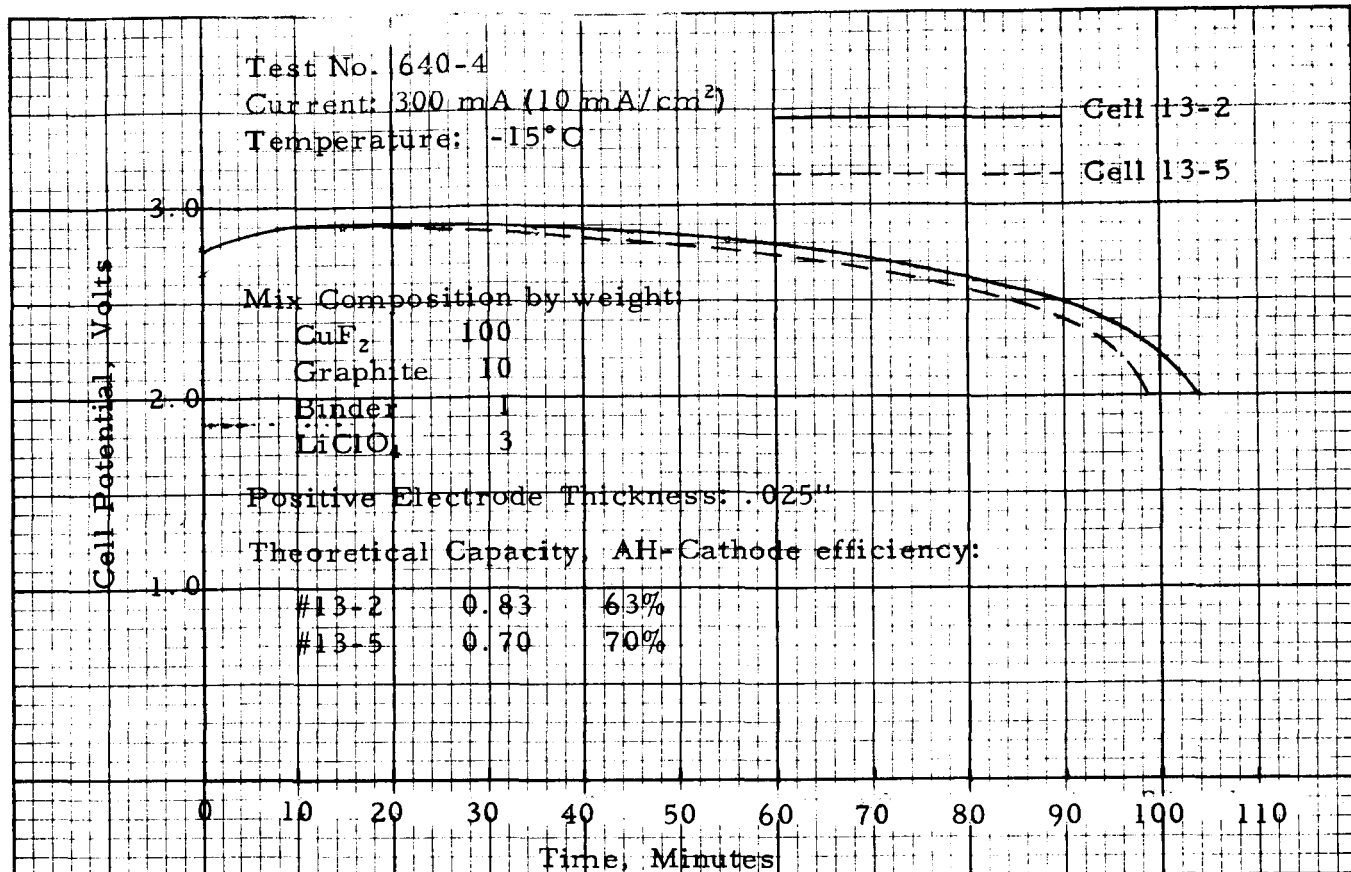
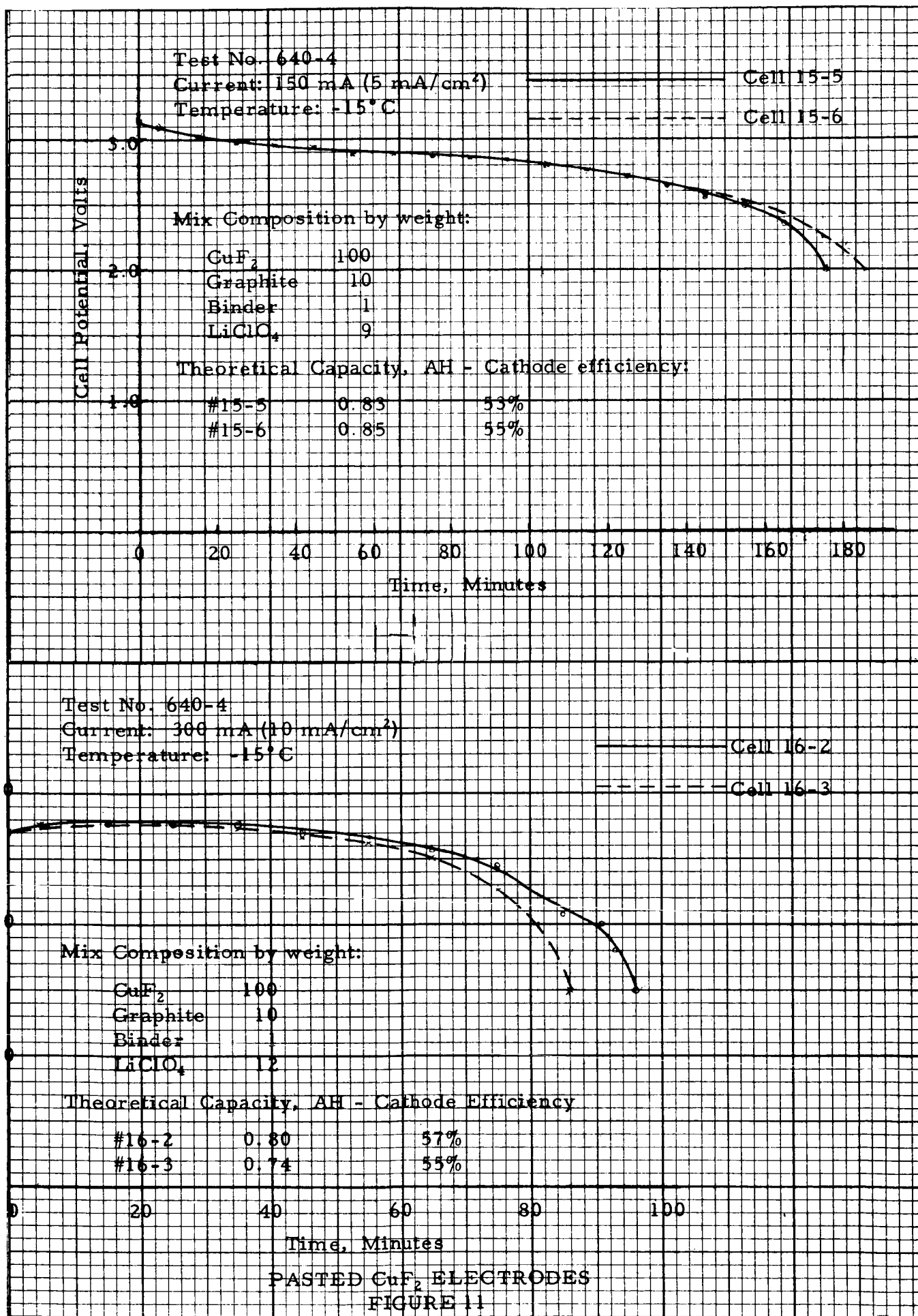
PASTED CuF₂ ELECTRODES

FIGURE 10



3.2.2. Filter-Mat CuF_2 Electrode Tests

Study of the filter-mat electrode construction described in previous reports (see, for example, NASA CR-54803, pages 67 to 72) was continued during the past quarter by constructing and discharging electrodes prepared with various CuF_2 -graphite-paper fiber ratios. The ratio of the quantity of CuF_2 to cross-sectional electrode area was held constant at 1.79g CuF_2 /square inch of electrode area, while the weights of graphite and paper fiber were adjusted to give the desired mat composition. As in earlier tests, the CuF_2 electrodes had a cross-sectional area of 15 cm^2 and were assembled in three-plate cells with 0.03 inch microporous rubber separation and two lithium negative electrodes. The test assembly was completed by heat-sealing the cell components into a polyethylene envelope, and supporting the cells between wood blocks to provide lateral compression during discharge. The test cell components before assembly are pictured in Figure 12, page 36, while a group of four cells in the cell discharge "test rack" are shown in Figure 13, page 37.

The cells were activated with electrolyte consisting of 15 grams of LiClO_4 dissolved in 100 ml of propylene carbonate. The water content of the electrolyte solution as determined by Karl Fischer analysis was 140 ppm, while the CuF_2 used for preparing the filter-mat electrodes contained 1.2 percent of water determined by X-ray analysis.

After activation with the electrolyte solution, the cells were allowed to stand on open circuit for four hours, and were then put in discharge at 35°C thru 200 and 100 ohm loads. Construction data, and performance of the cells to a final voltage of 2.5 volts are presented in Tables XI and XII, pages 38 and 39; voltage-time data for the 48 cells discharged in this test series are presented in Figures 14 to 25 , pages 40 to 51.

Inspection of the cells at the end of discharge showed that in a number of cells, the CuF_2 electrode matrix had not been completely penetrated by the electrolyte solution. This condition was more prevalent in cells which had a high weight ratio of graphite to paper fiber in the CuF_2 electrode matrix. This condition, which is in part reflected by the varying electrolyte volume figures presented in Table XI , page 38 , was most likely caused by the poor wetting properties of the graphite. The results of the discharge tests obtained must, therefore, be viewed with due regard to the reservations discussed above.

With the 200 Ω discharge loads (ca. 0.5 mA/cm²) efficiency of the CuF₂ electrode was as high as 81 percent to a final cell voltage of 2.5 volts, while with 100 Ω load the highest efficiency figure recorded was 65 percent. Optimum fiber concentration appeared to be in the range of 4 to 8 grams per 100g of CuF₂, and the best graphite level was in the range of 8-20 grams per 100g of CuF₂. However, some efficient discharges did occur in the cells having additive levels outside the above described ranges.

Further studies of electrode composition will be conducted, since considerable improvement in the theoretical energy-to-weight ratio can be achieved by lowering the fraction of inert additives in the CuF₂ electrode. To avoid the complications which result from non-uniform wetting of the matrix, vacuum impregnation of the electrodes with the electrolyte solution will be studied.

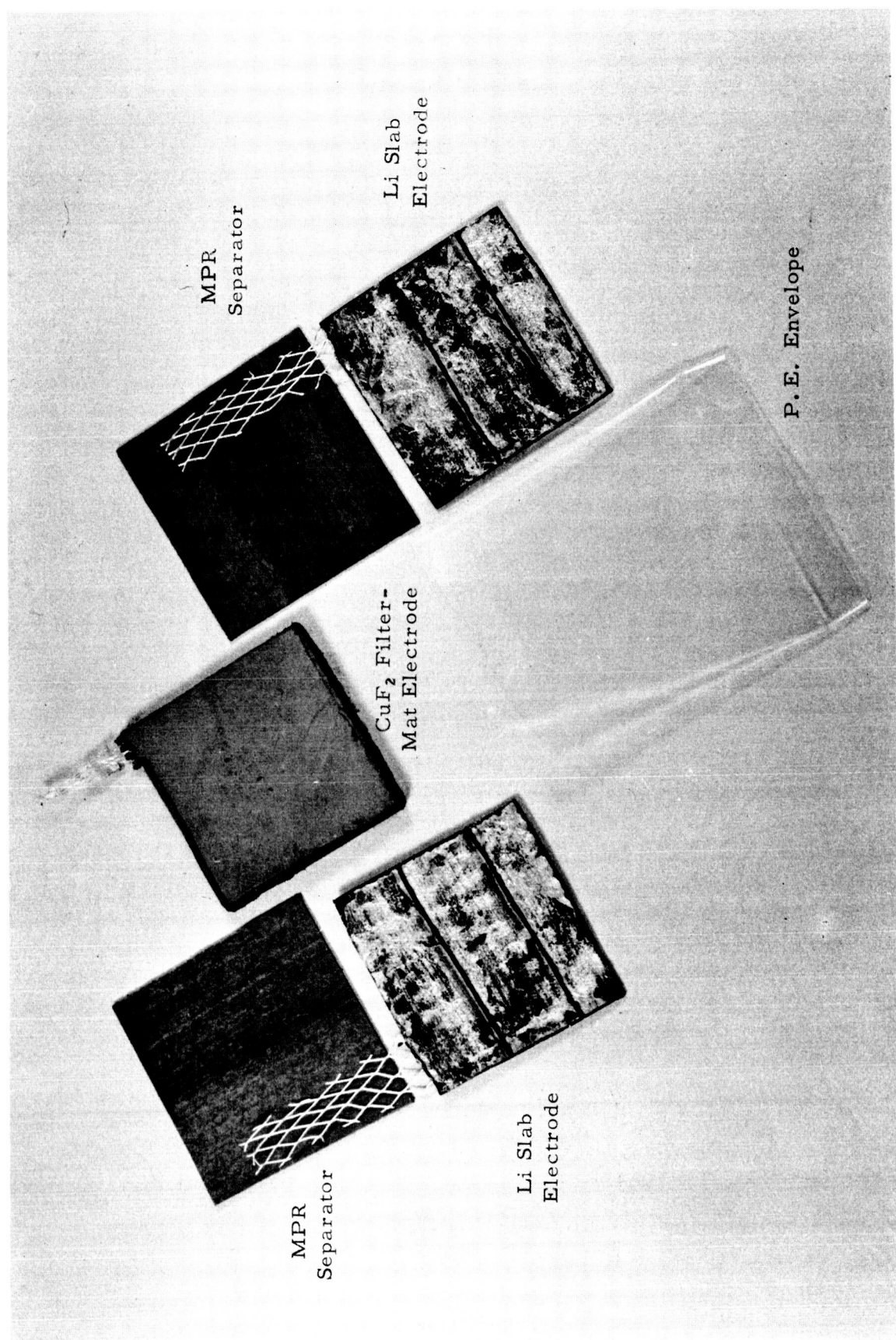
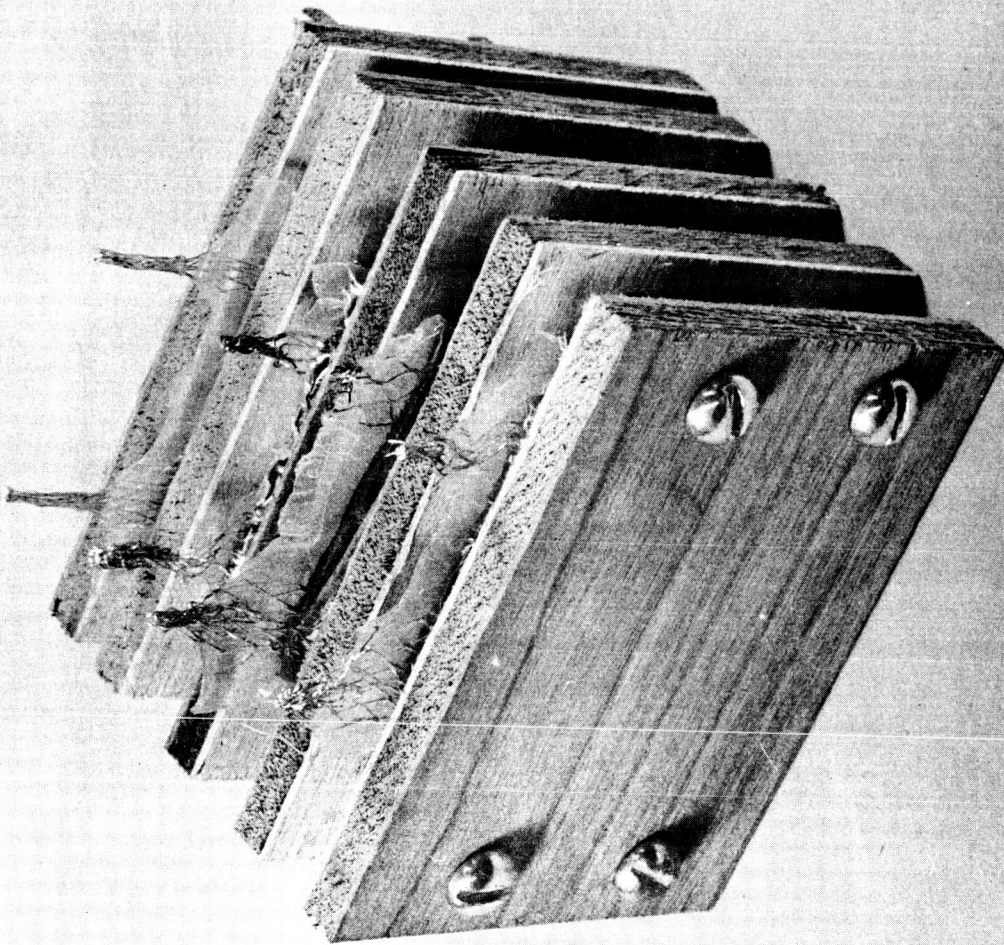
CuF₂-Li TEST CELL COMPONENTS

FIGURE 12



CELL DISCHARGE TEST RACK

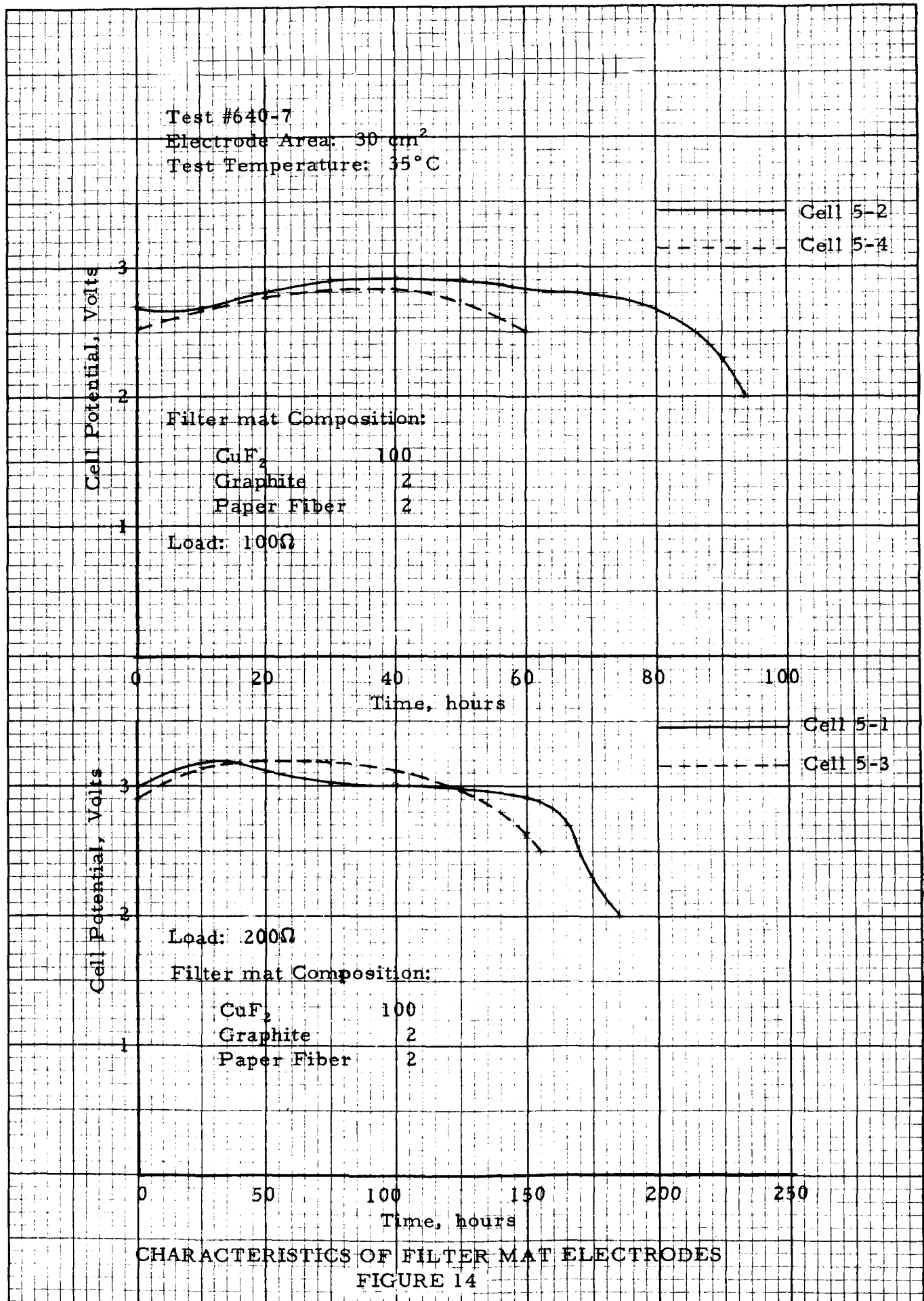
FIGURE 13

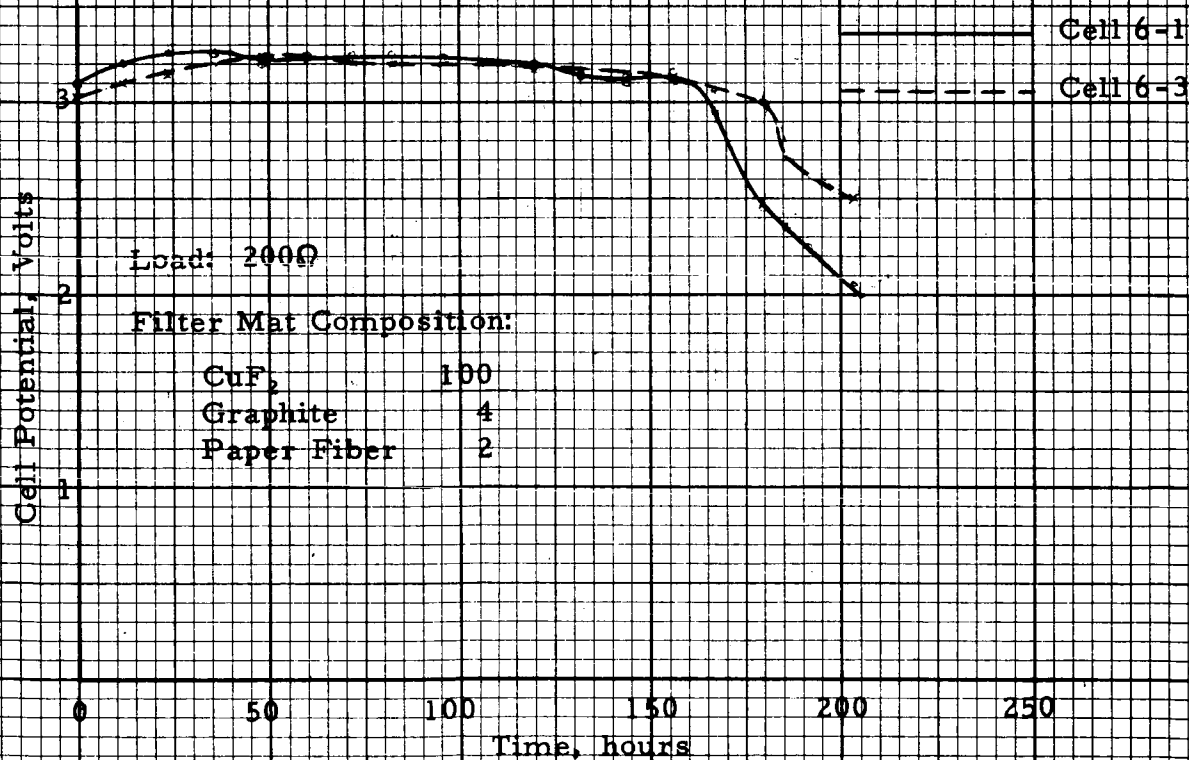
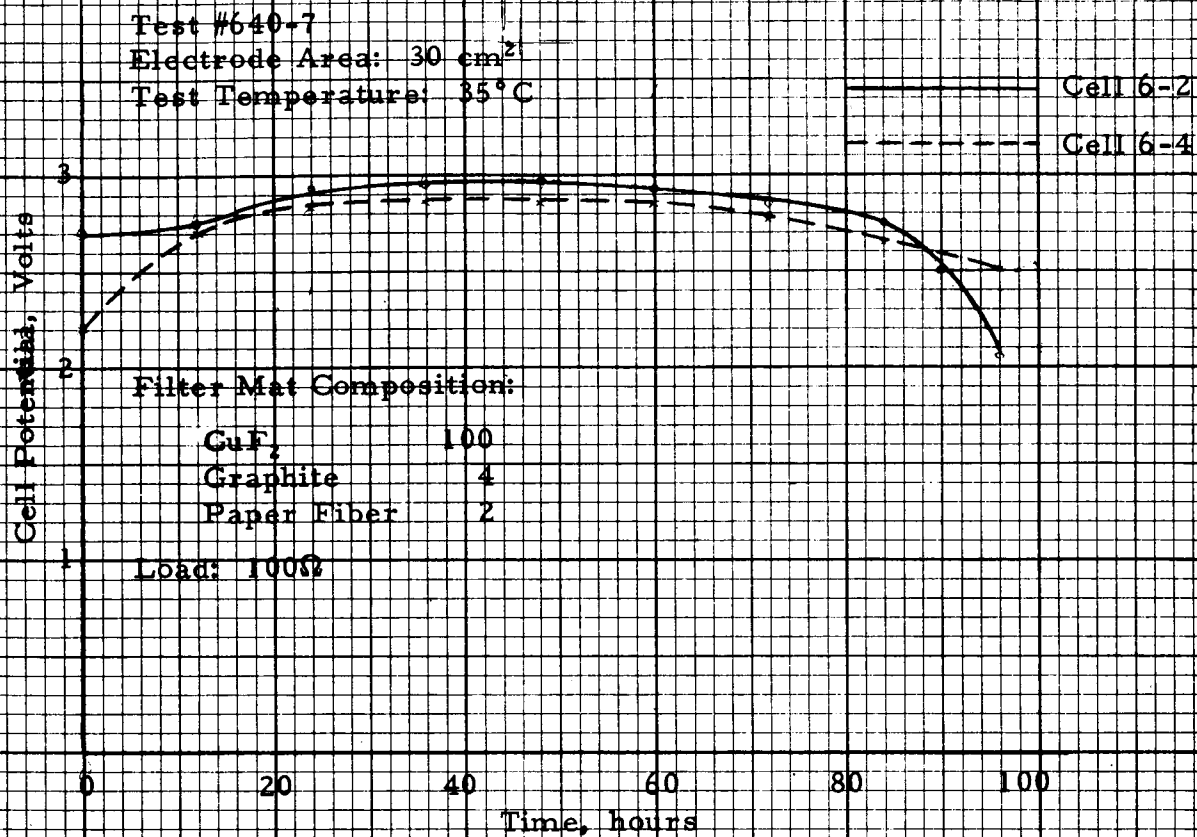
TABLE XI
FILTER-MAT CuF₂ ELECTRODE TESTS
 200 Ω Discharge

Cell No.	Additive grams/100g CuF ₂		Theoretical Capacity, AH	Electrolyte Volume, cc	Average Discharge Potential	A-min/gram Positive Electrode	CuF ₂ Efficiency, %
	Graphite	Paper Fiber					
5-1	2	2	4.42	5.0	3.01	16.7	57
5-3	2	2	4.13	6.5	3.05	16.3	53
6-1	4	2	3.92	6.0	3.13	20.6	72
6-3	4	2	4.54	5.5	3.10	19.4	67
7-1	8	2	3.75	6.0	3.11	18.0	65
7-3	8	2	3.76	5.5	3.10	18.9	65
8-1	14	2	3.92	6.0	3.11	18.1	69
8-3	14	2	3.80	7.5	2.82	15.1	58
9-1	4	4	5.05	5.5	---	---	--
9-3	4	4	3.67	4.0	3.10	17.3	61
10-1	8	4	4.34	6.5	3.00	17.8	63
10-3	8	4	4.75	5.5	3.17	15.6	57
11-1	14	4	3.90	6.5	3.14	18.9	73
11-3	14	4	3.41	7.0	3.14	19.6	76
12-1	20	4	4.44	6.5	3.12	17.9	72
12-3	20	4	4.13	6.5	3.18	20.1	81
13-1	4	8	4.35	6.0	3.14	18.9	69
13-3	4	8	5.03	6.5	3.08	18.5	67
14-1	8	8	4.13	6.0	3.11	19.1	72
14-3	8	8	3.70	5.5	3.12	20.6	78
15-1	14	8	4.19	7.0	3.13	20.3	81
15-3	14	8	3.26	5.0	3.00	15.2	61
16-1	20	8	3.98	7.0	3.03	14.2	59
16-3	20	8	4.28	5.0	3.05	9.5	40

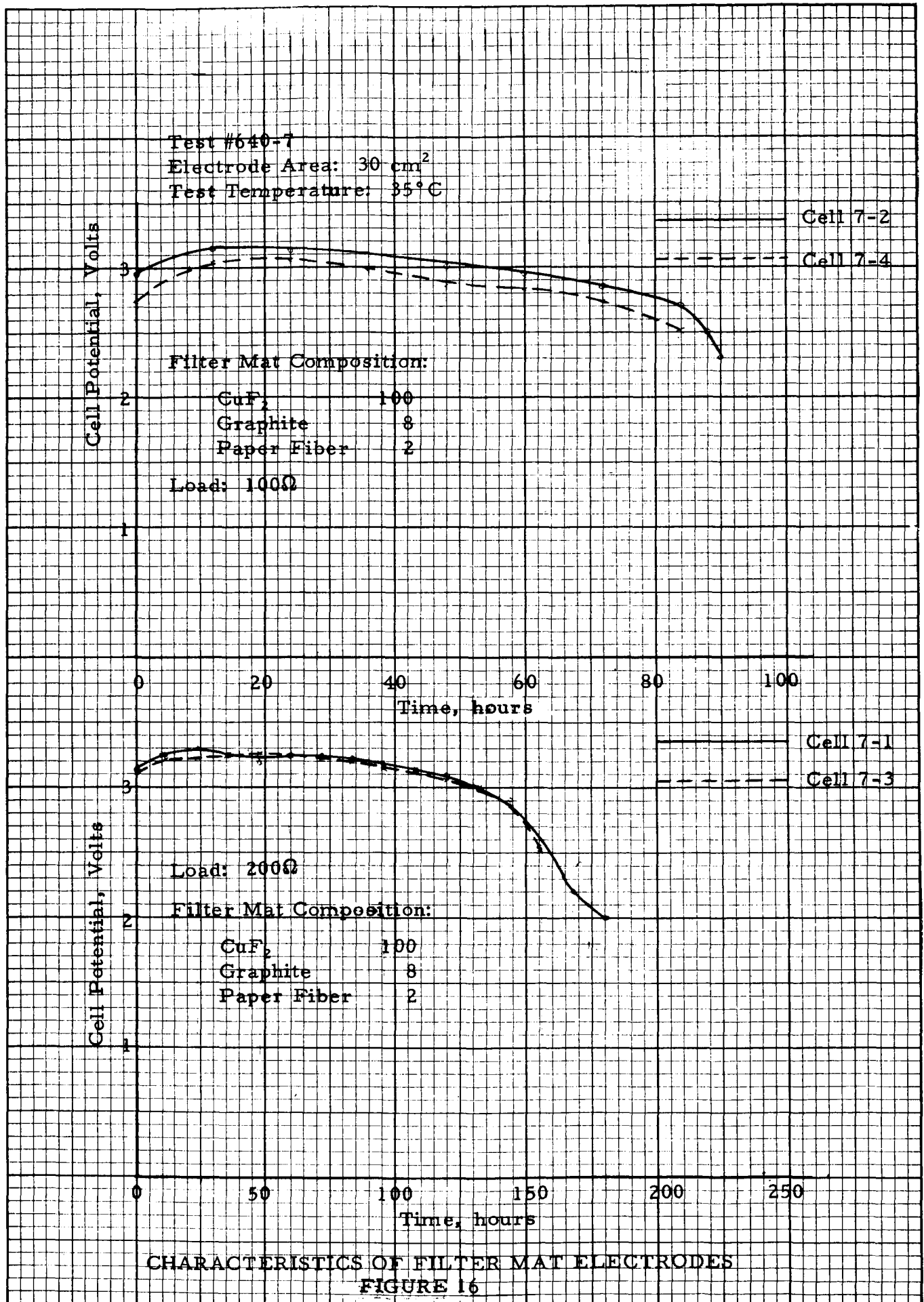
TABLE XII
FILTER-MAT CuF₂ ELECTRODE TESTS
 100 Ω Discharge

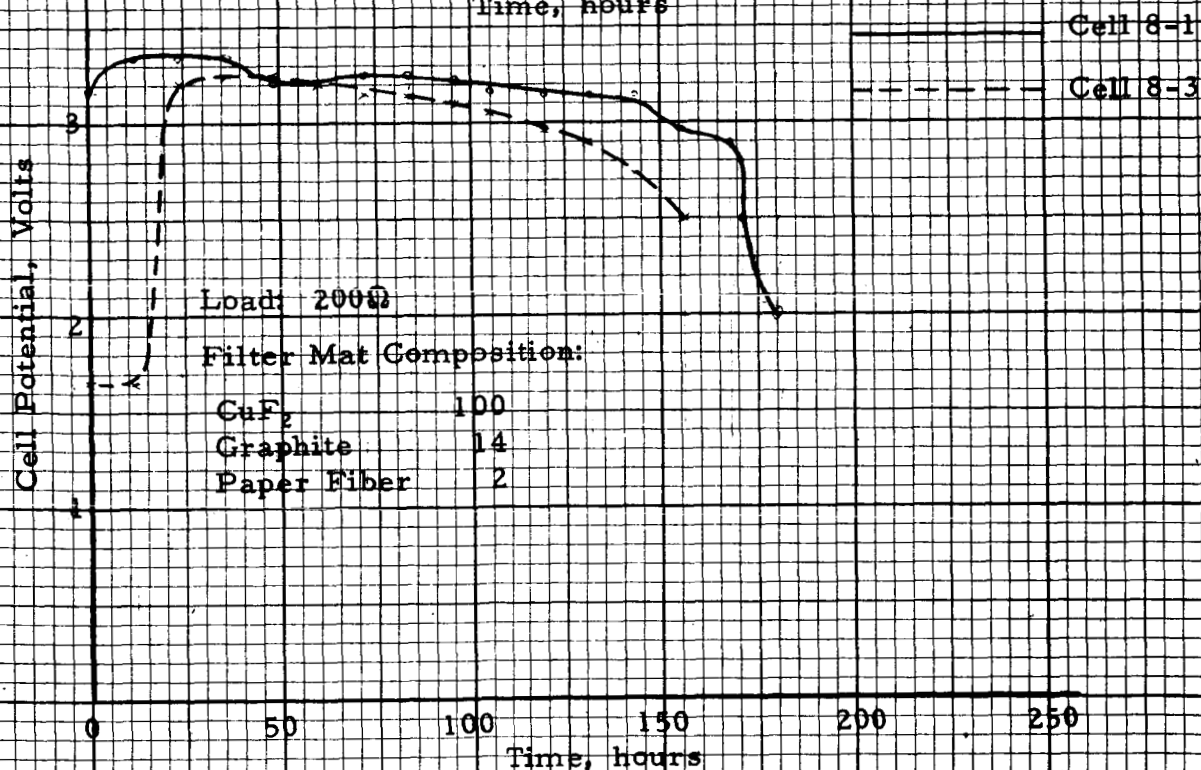
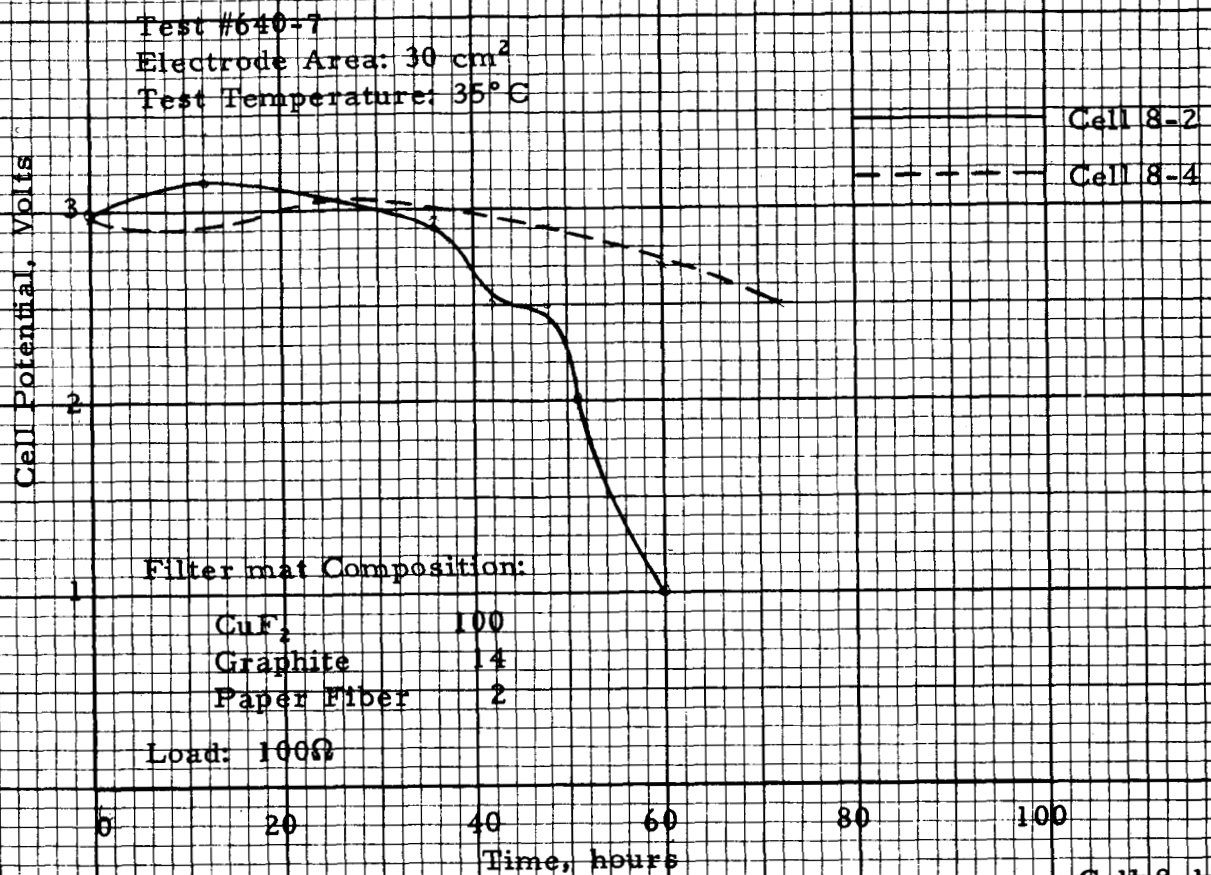
Cell No.	Additive grams/100g CuF ₂		Theoretical Capacity, AH	Electrolyte Volume, cc	Average Discharge Potential	A-min/gram. Positive Electrode	CuF ₂ Efficiency, %
	Graphite	Paper Fiber					
5-2	2	2	4.45	5.5	2.79	15.5	53
5-4	2	2	3.55	5.5	2.69	13.2	45
6-2	4	2	4.16	5.5	2.81	17.5	61
6-4	4	2	4.10	5.5	2.69	16.9	59
7-2	8	2	3.76	5.5	2.81	17.9	65
7-4	8	2	4.00	5.0	2.84	16.3	59
8-2	14	2	3.94	5.0	2.92	8.2	31
8-4	14	2	3.71	5.0	2.88	14.0	53
9-2	4	4	4.61	6.0	2.90	15.6	55
9-4	4	4	3.29	5.0	2.84	16.7	61
10-2	8	4	3.39	5.0	3.02	14.4	53
10-4	8	4	3.77	6.5	2.90	17.3	63
11-2	14	4	4.17	5.5	3.00	13.1	50
11-4	14	4	3.67	5.5	2.88	14.0	54
12-2	20	4	3.86	5.0	2.97	12.6	51
12-4	20	4	4.47	7.0	2.91	12.0	48
13-2	4	8	4.85	6.0	2.67	6.1	22
13-4	4	8	4.31	6.0	2.72	13.9	51
14-2	8	8	3.38	5.0	2.94	13.7	52
14-4	8	8	3.52	6.0	2.82	14.9	57
15-2	14	8	3.58	5.5	2.92	11.0	44
15-4	14	8	4.18	6.0	2.78	14.8	59
16-2	20	8	4.23	5.0	---	---	--
16-4	20	8	4.28	7.0	2.88	12.4	51





CHARACTERISTICS OF FILTER MAT ELECTRODES
FIGURE 15





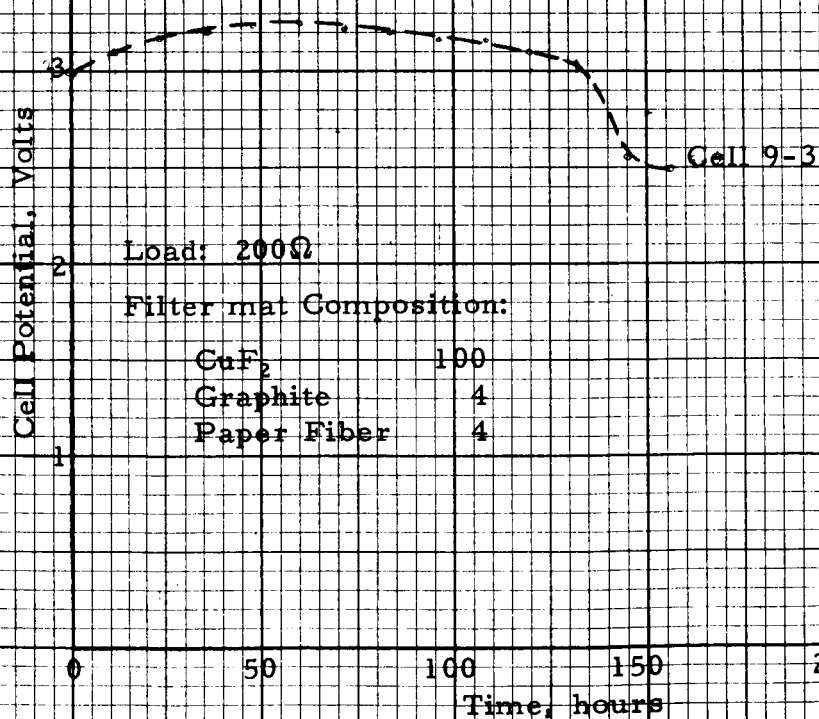
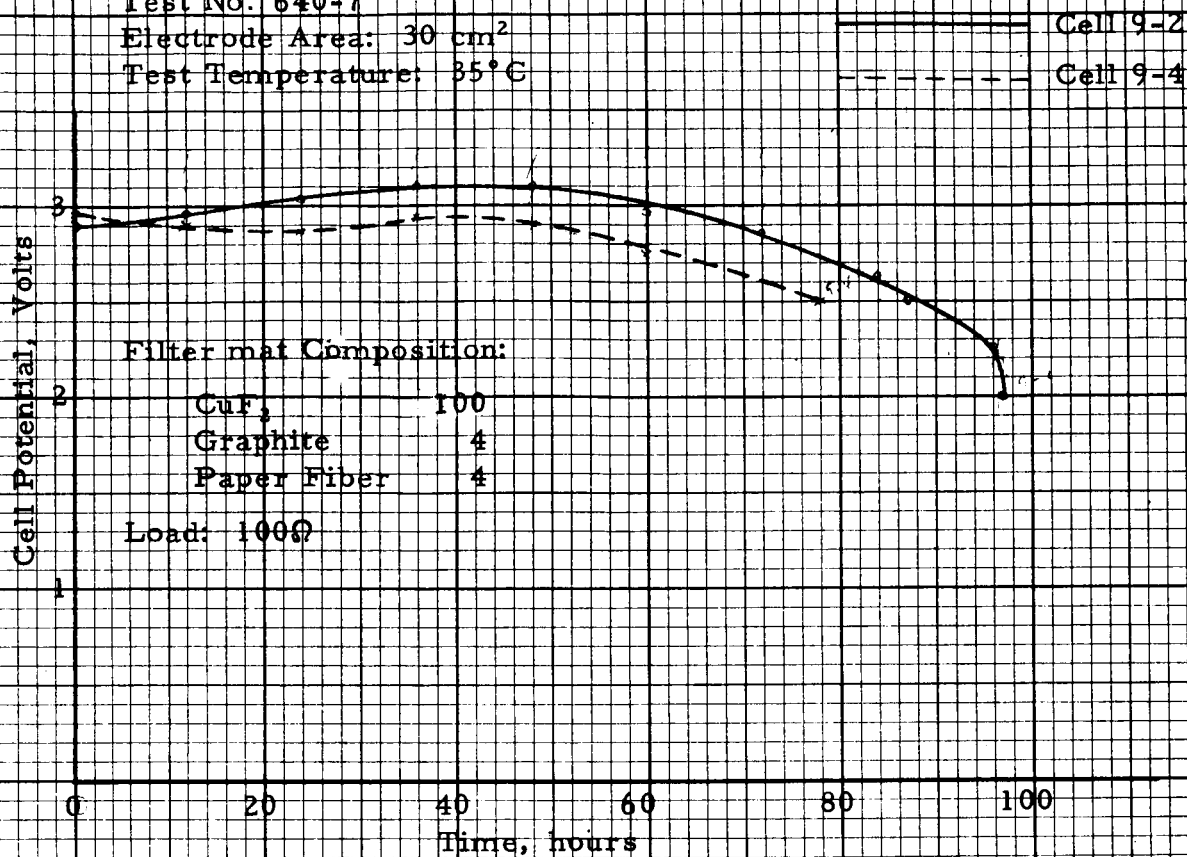
CHARACTERISTICS OF FILTER MAT ELECTRODES

FIGURE 17

Test No. 640-7

Electrode Area: 30 cm²

Test Temperature: 35°C

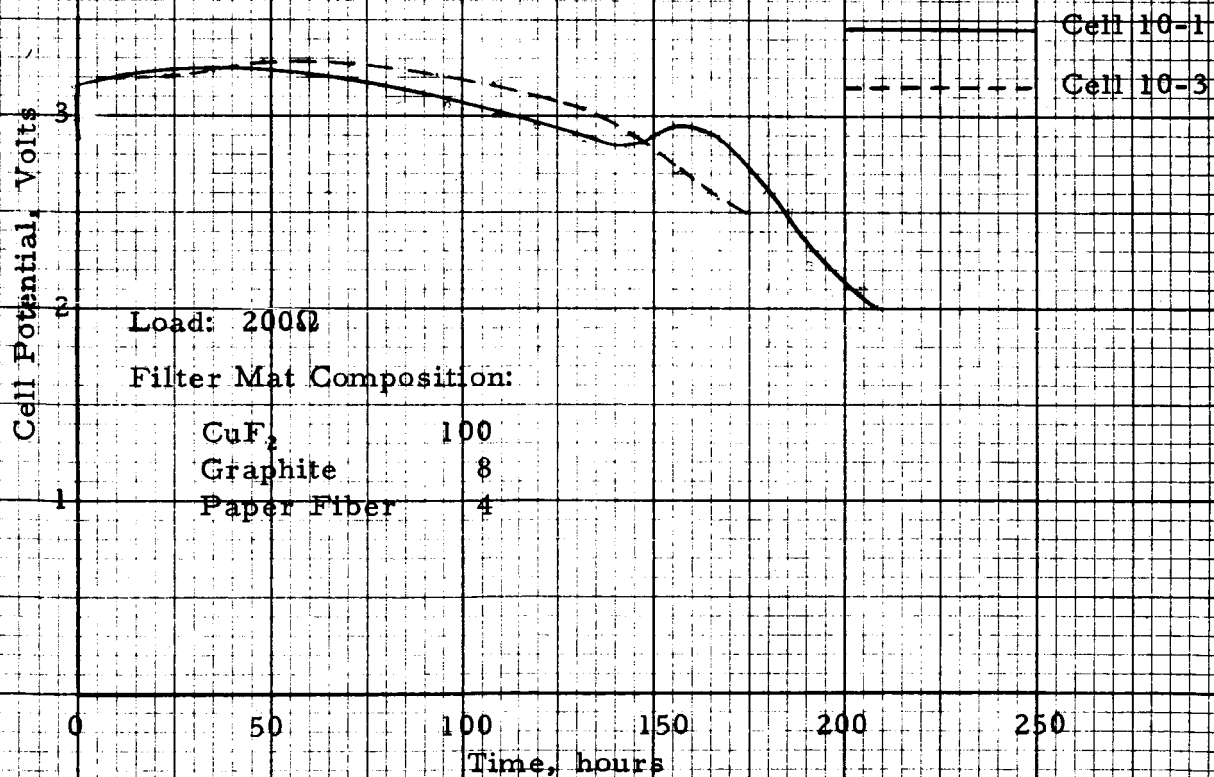
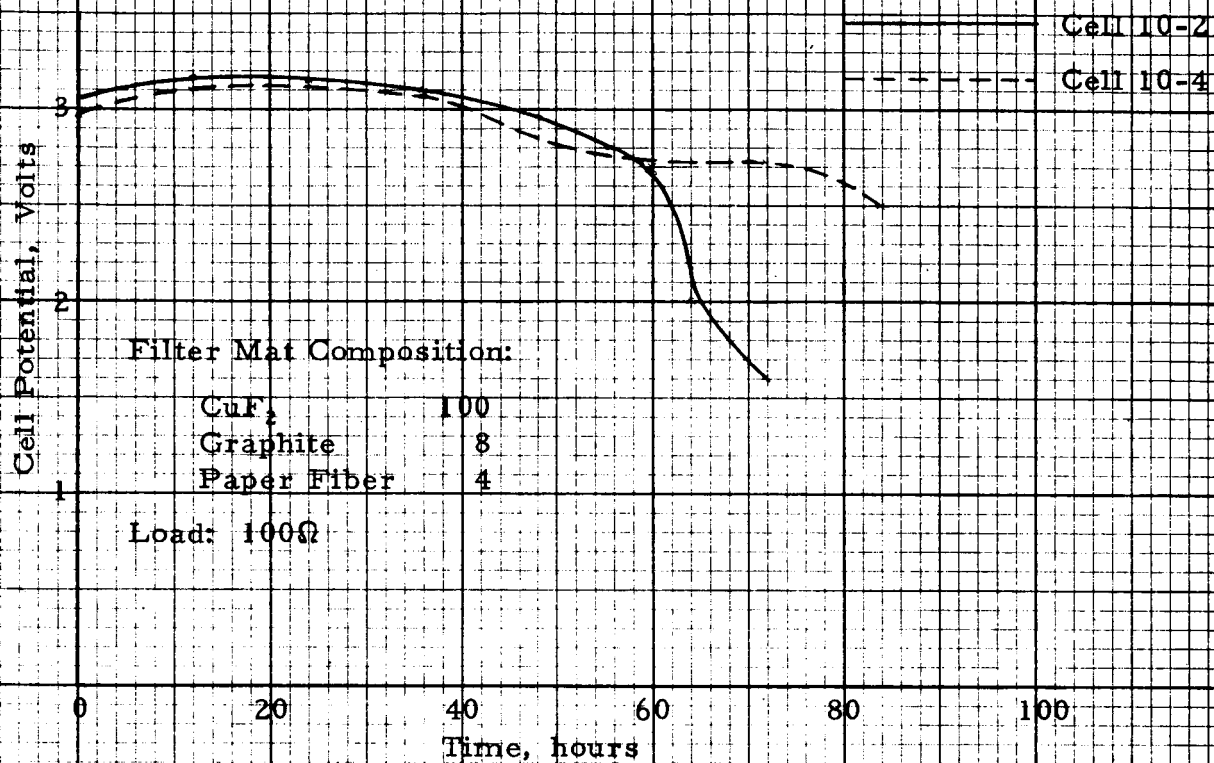


CHARACTERISTICS OF FILTER MAT ELECTRODES
FIGURE 18

Test #640-7

Electrode Area: 30 cm²

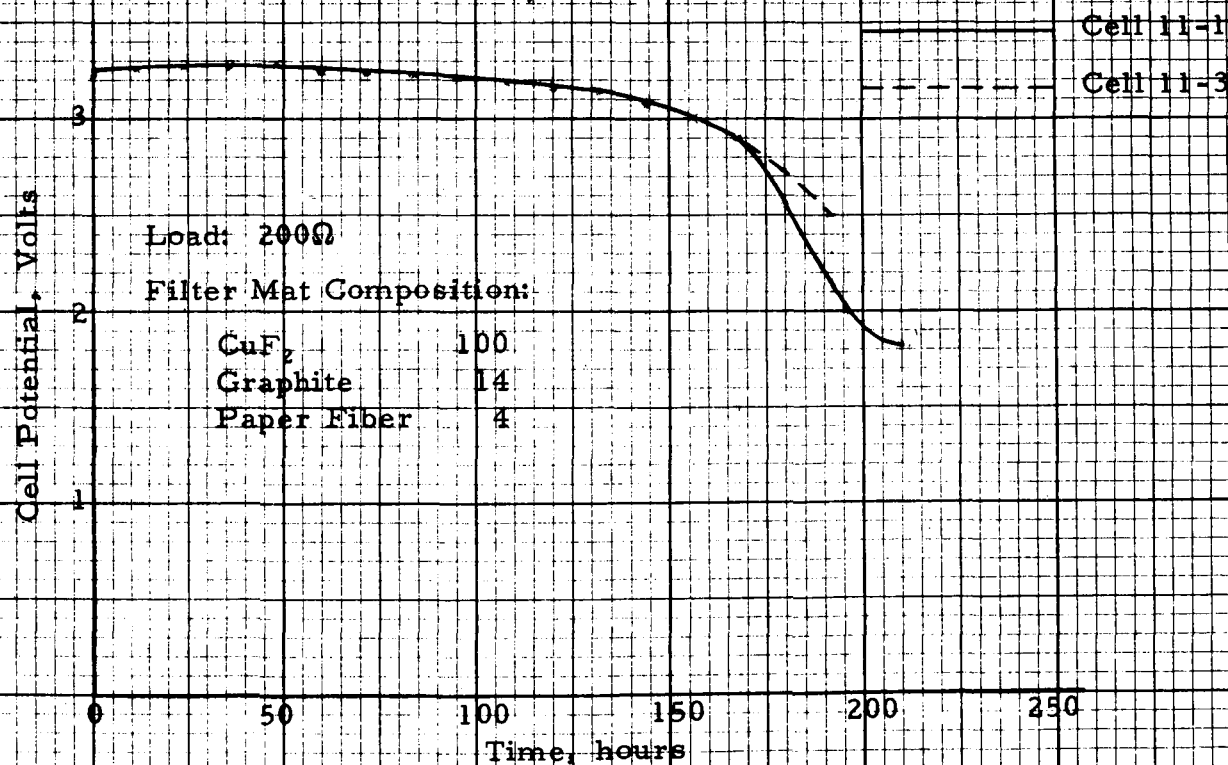
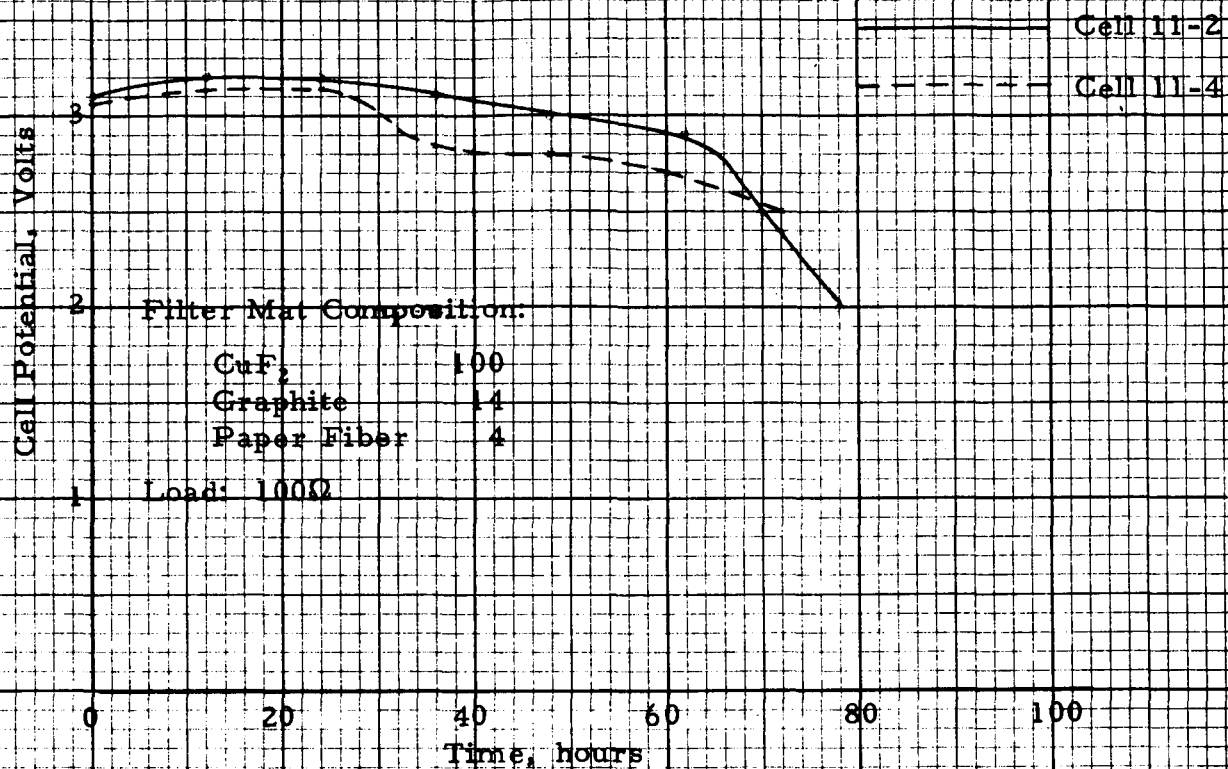
Test Temperature: 35°C



CHARACTERISTICS OF FILTER MAT ELECTRODES

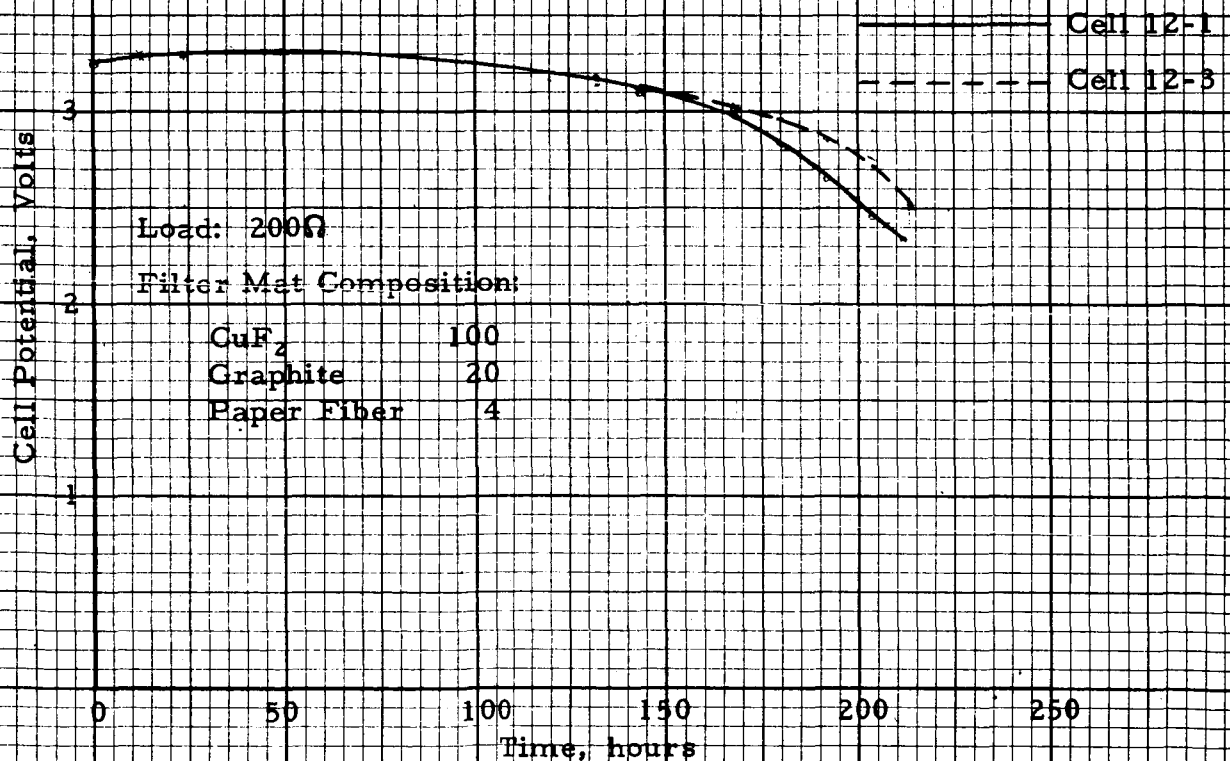
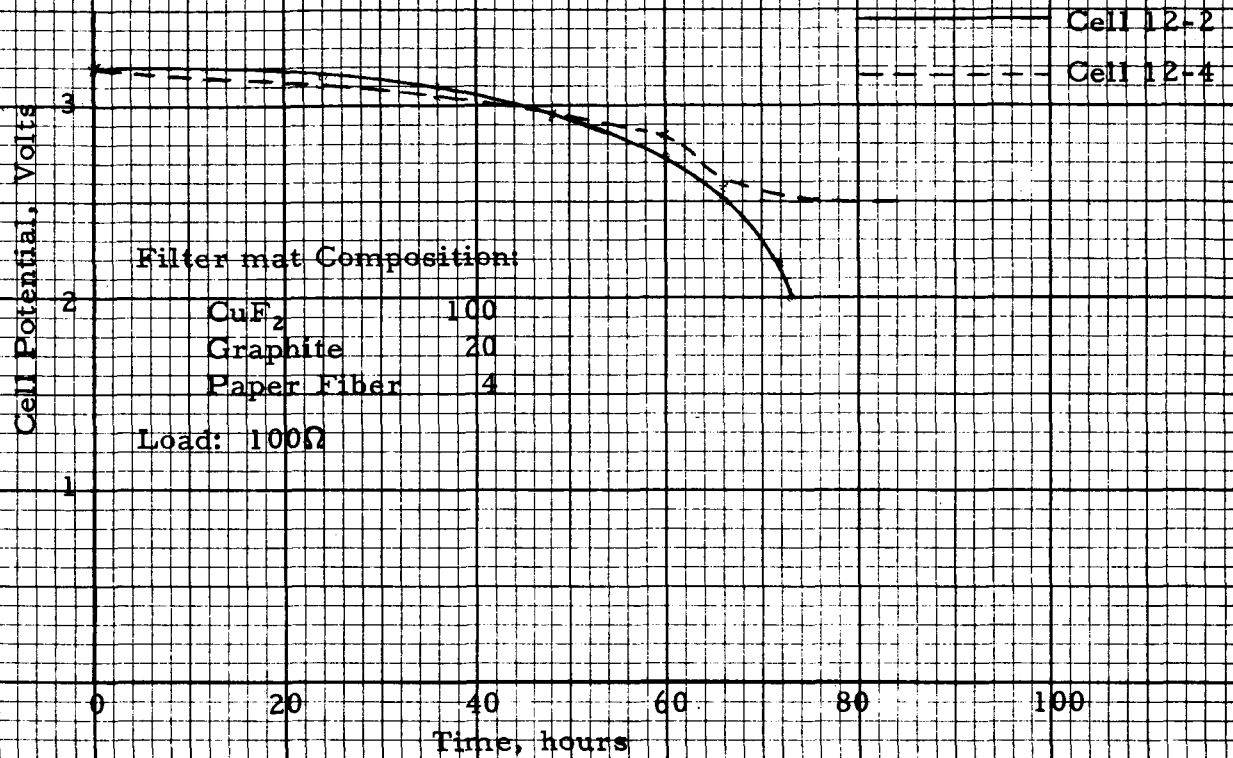
FIGURE 19

Test #640-7
 Electrode Area: 30 cm²
 Test Temperature: 35°C



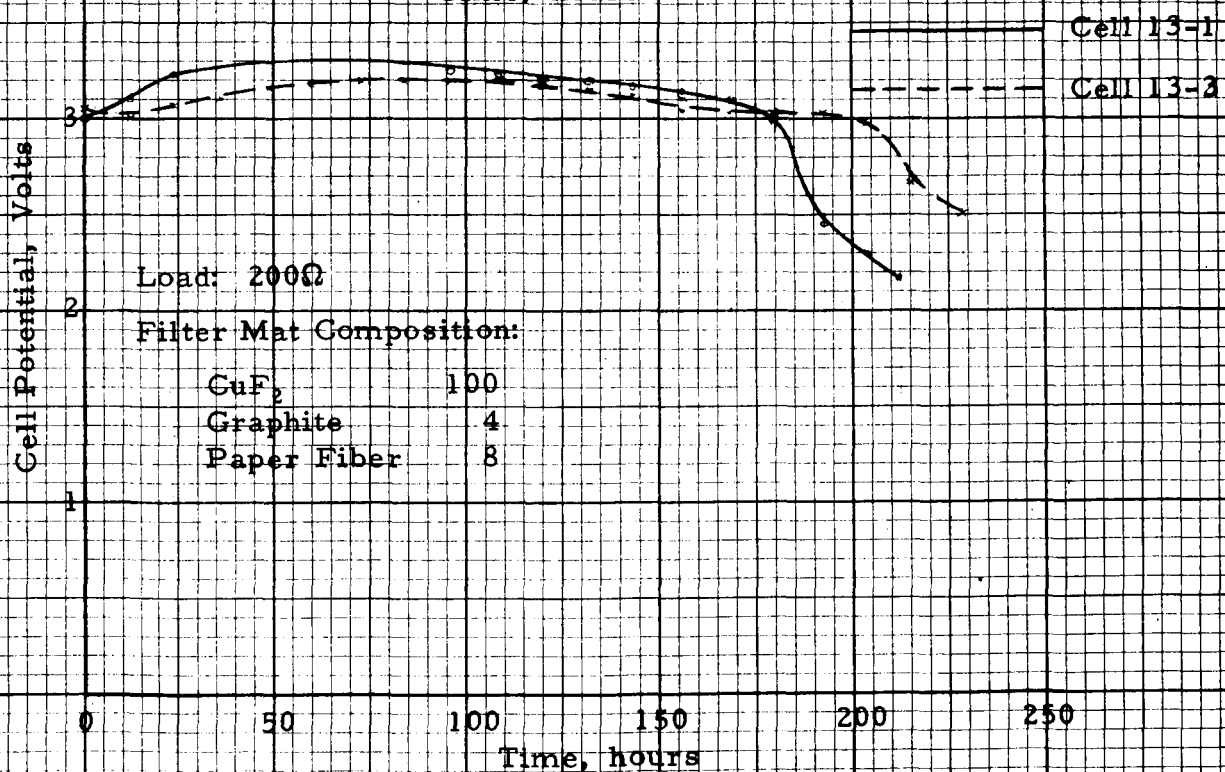
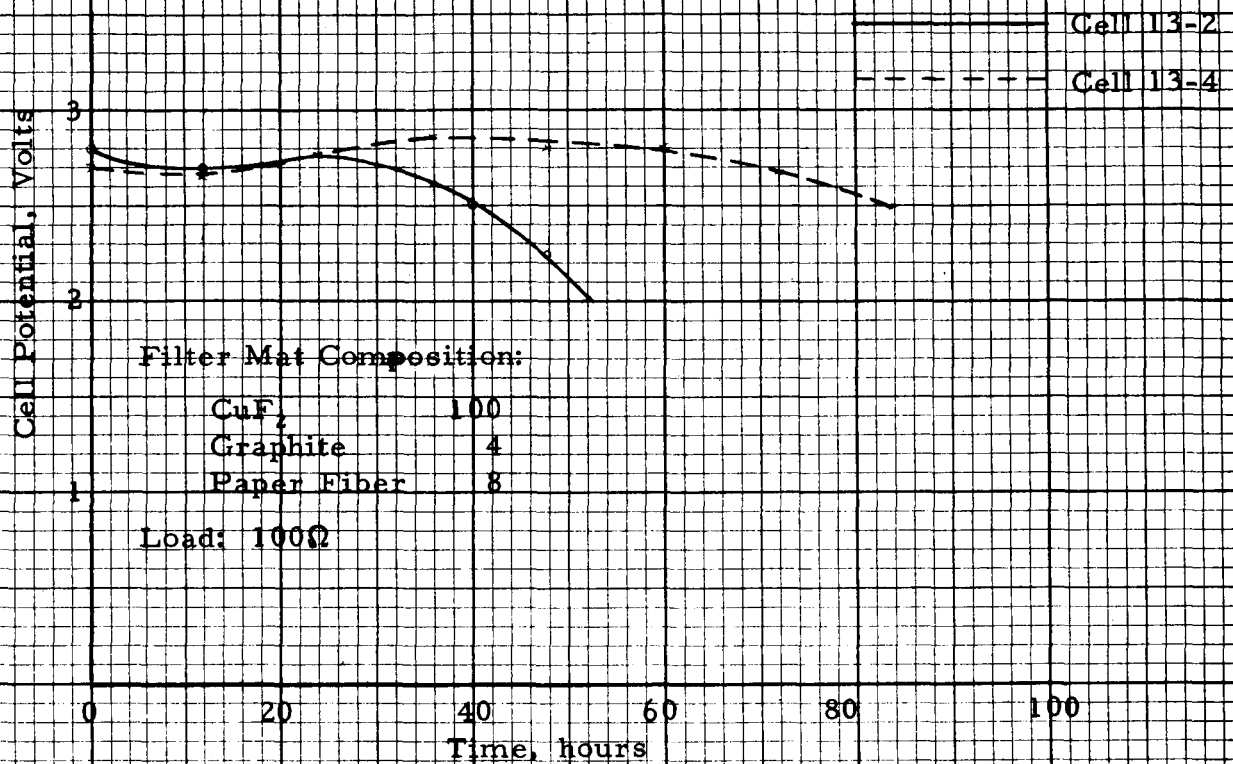
CHARACTERISTICS OF FILTER MAT ELECTRODES
 FIGURE 20

Test #640-7
 Electrode Area: 30 cm²
 Test Temperature: 35°C



CHARACTERISTICS OF FILTER MAT ELECTRODES
 FIGURE 21

Test #640-7
 Electrode Area: 30 cm²
 Test Temperature: 35°C

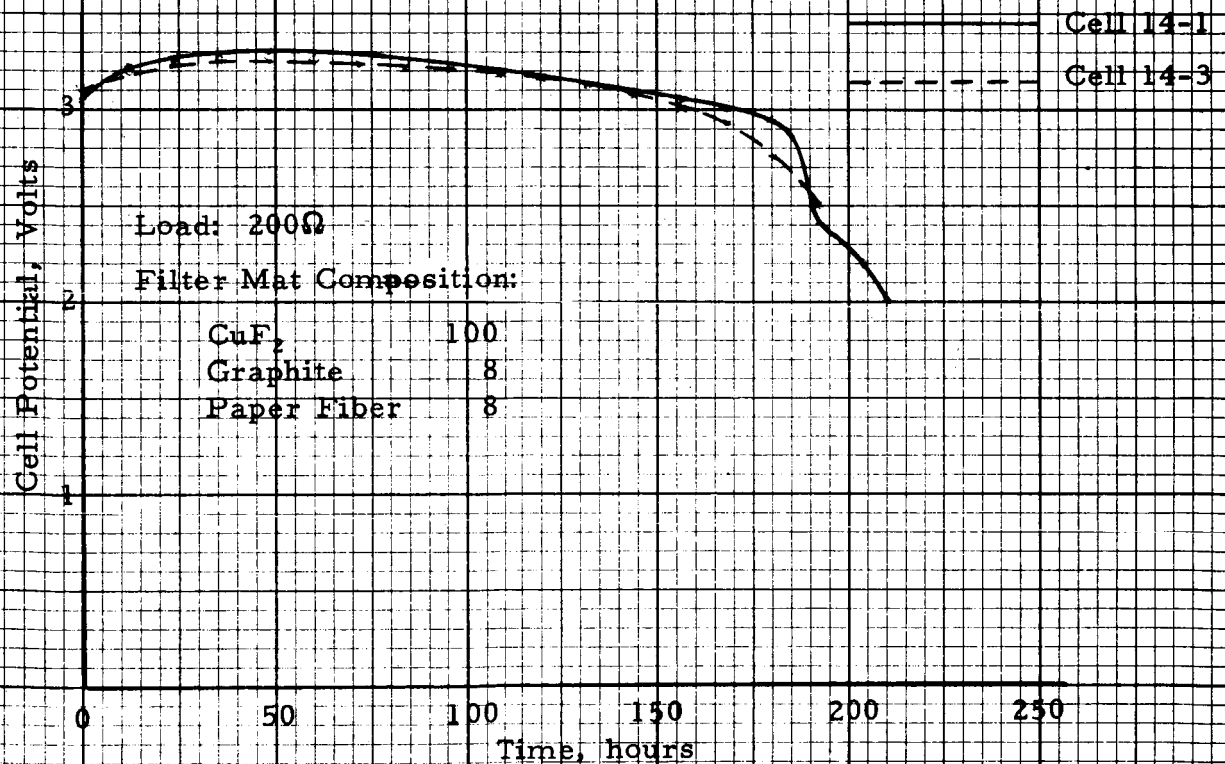
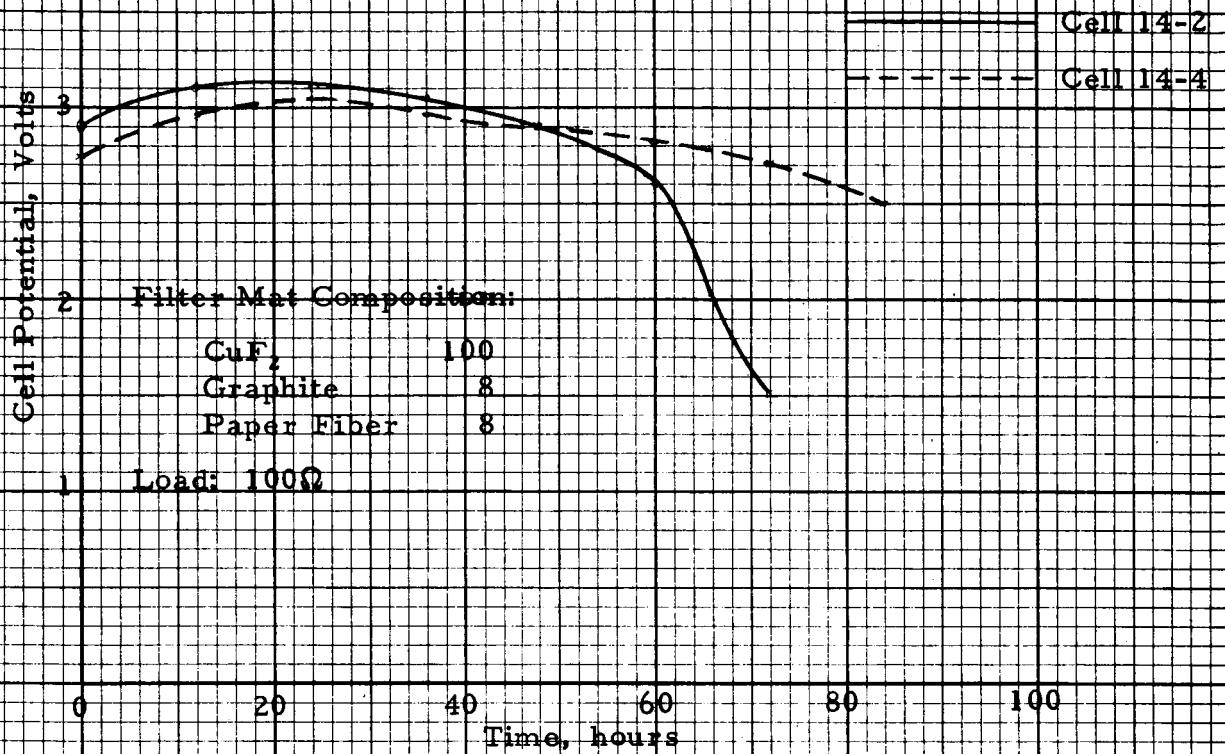


CHARACTERISTICS OF FILTER MAT ELECTRODES
 FIGURE 22

Test #640-7

Electrode Area: 30 cm²

Test Temperature: 35°C

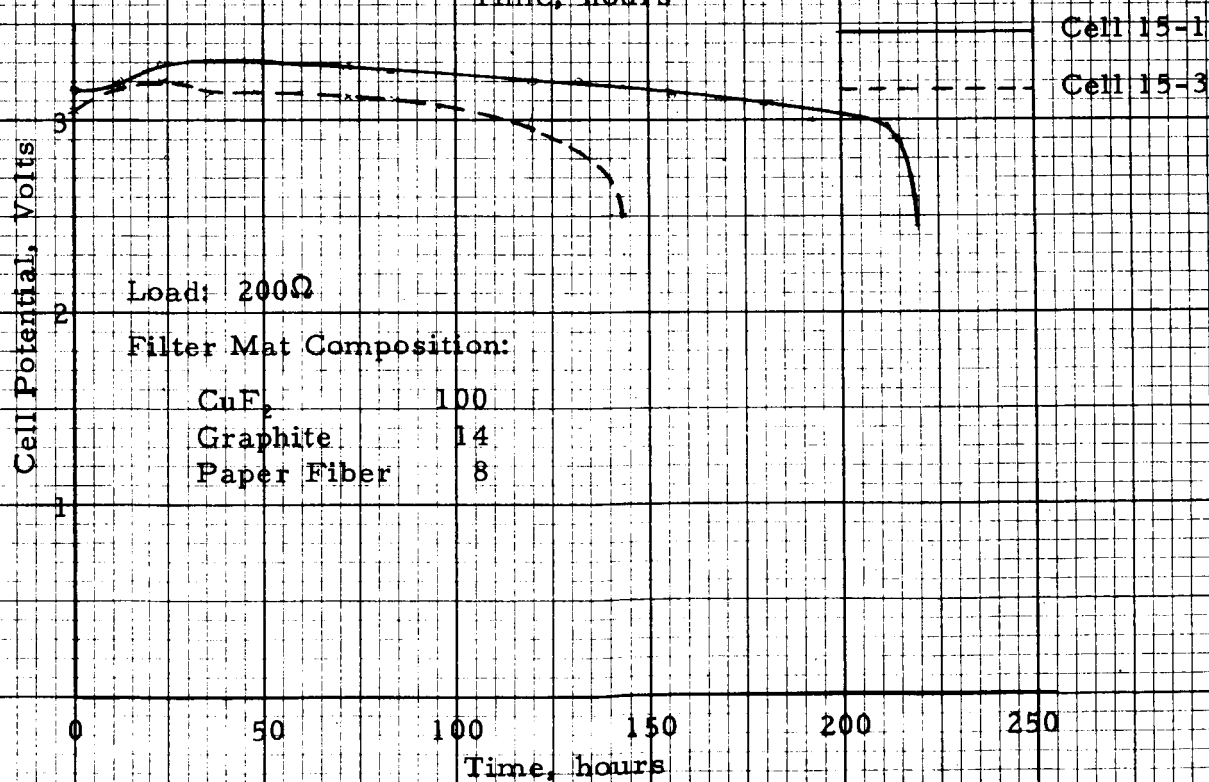
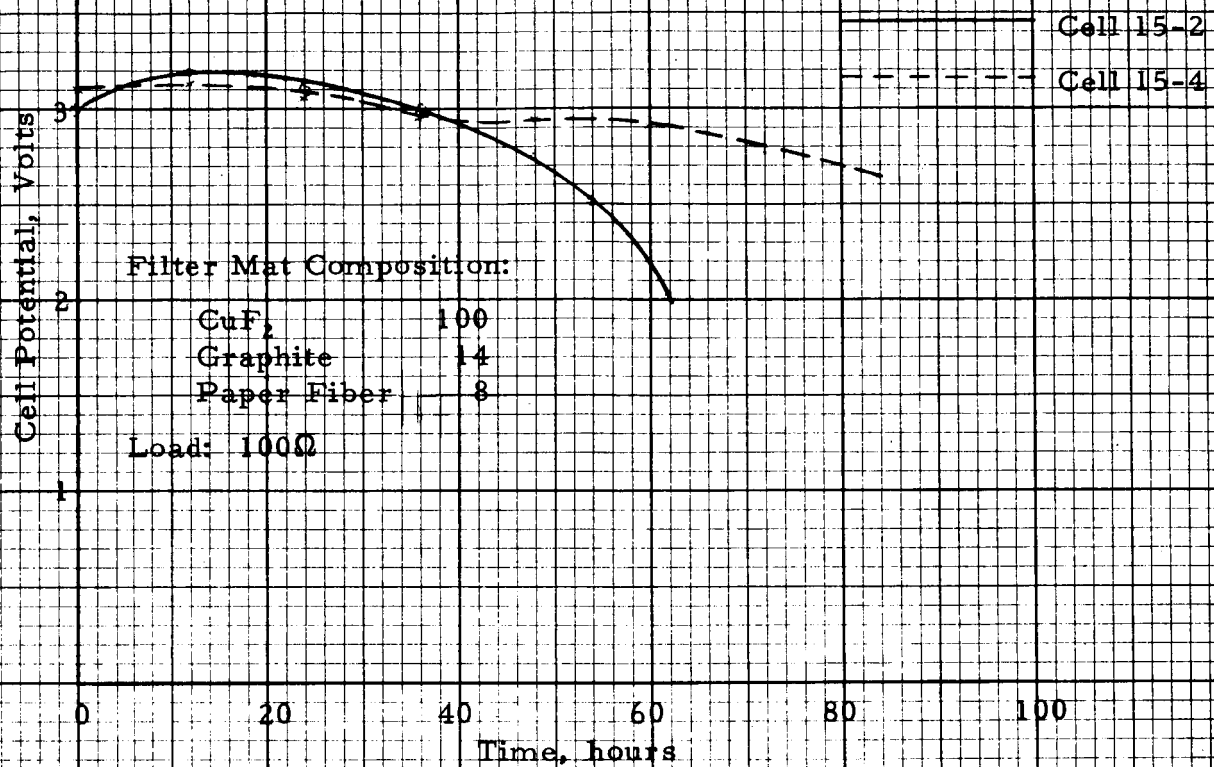


CHARACTERISTICS OF FILTER MAT ELECTRODES
FIGURE 23

Test #640-7

Electrode Area: 30 cm²

Test Temperature: 35°C



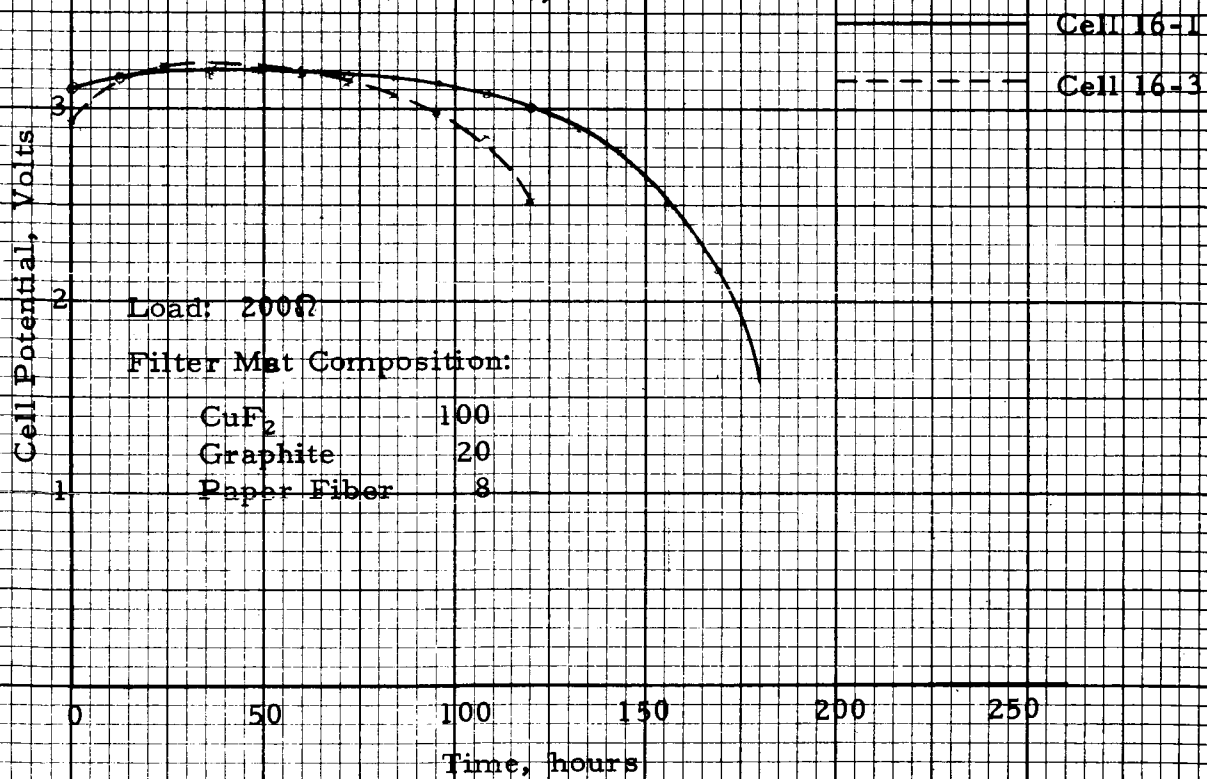
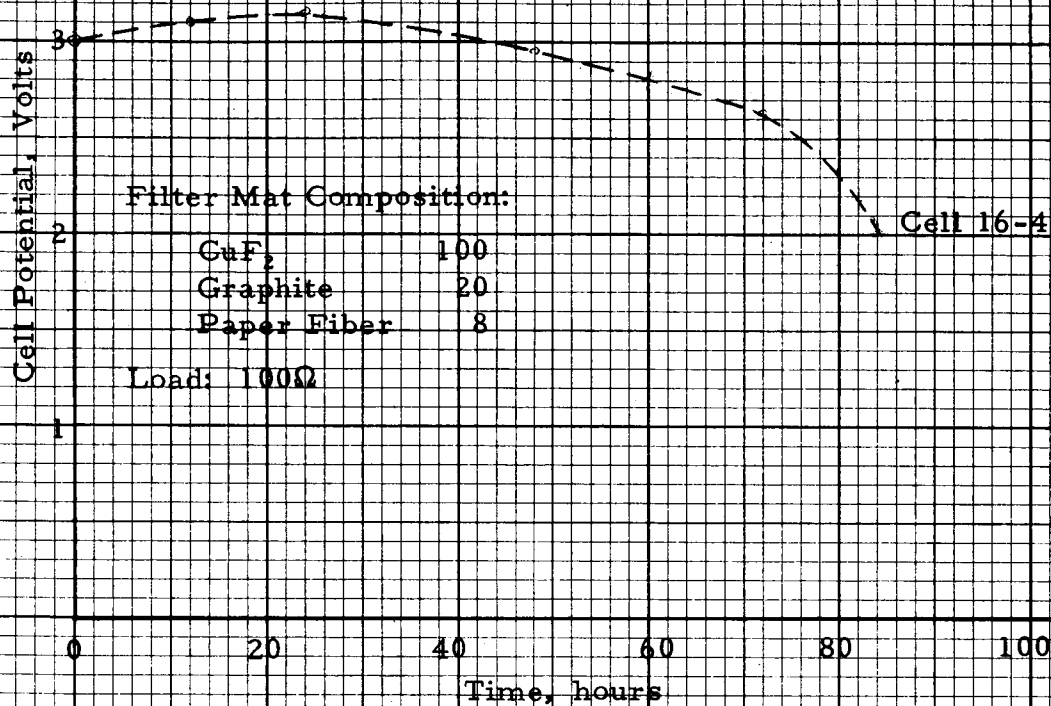
CHARACTERISTICS OF FILTER MAT ELECTRODES

FIGURE 24

Test #640-7

Electrode Area: 30 cm²

Test Temperature: 35°C



CHARACTERISTICS OF FILTER MAT ELECTRODES
FIGURE 25

3.3. CELL SYSTEMS STUDIES

Although it has been evident that, in their present state of development, the CuF_2 -Li cells would have very poor shelf life after activation with the electrolyte, a program for the study of the rate of self-discharge in these cells was initiated. This was done in order to both establish the actual rate of degradation of the cells during the stand period, and to identify the factors which contribute most severely to the capacity decay.

The present test series was performed with cells having construction similar to that used for the prototype cells built under the previous contract. The cells have one filter-mat positive electrode (82 percent CuF_2 , 12 percent graphite, 6 percent paper pulp. 1.5" x 1.5", 4-5 AH theoretical), and two lithium sheet negative electrodes; the separation was 0.03" microporous rubber, and propylene carbonate electrolyte was used (15g LiClO_4 /100 ml PC).

The water content in the electrolyte used in this test series was 500 ppm, including water introduced with the solute. For one group of cells, CuF_2 having a water content of 2.8 percent (X-ray analysis) was used, while for the second group the same material was dried at 70° for 16 hours at full vacuum (100-200 microns). The water content in the treated material was not determined, but other drying tests on the same material indicate that the water content may have been in the range of 1-2 percent after drying.

After the test cells had been filled with the electrolyte, four cells from each group were put in discharge through 200 Ω resistors at 35°C, while the remaining cells were stored at 35°, -15°, and -55°C. It was originally intended to discharge groups of two cells from each of the test environments after a stand of 1-month, 3-months, and 6-months; however, these intervals were changed to 2-weeks, 4-weeks, and 6-weeks after a rapid open circuit potential decay was observed in many cells after a stand of only 2-weeks.

Construction and discharge data for the test cells are summarized in Tables XIII and XIV, pages 54 and 55, while the voltage-time data for the discharges are shown in Figures 26 to 32, pages 56 to 63. As expected, poor capacity retention was exhibited by the cells which had been on stand at 35°C. At -15° and -55°C, capacity retention was in the order of 80 percent after four weeks of stand for the cells built with the vacuum-dried CuF_2 . After six weeks of stand, less capacity was retained by cells at -15°C than at -55°C.

Inspection of the cells after discharge showed the lithium slab electrodes to be in good condition except for the reduction in thickness which results from discharge. The inside surfaces of the lithium electrodes (those facing the CuF_2 electrode) showed a green deposit which was more pronounced in cells which had shown poor shelf life.

Since the effect of water contamination on the solubility of CuF_2 in the electrolyte has been clearly established, improvement in shelf life should be possible by further reducing the water content of the cell construction materials. With the drying and water monitoring techniques presently available in this contractor's laboratory, the practical lower limits of water appear to be 0.1 percent in the CuF_2 , and 100 ppm in the electrolyte solution. If these levels of dryness do not prove to be adequate in providing an acceptable shelf life to the CuF_2 -Li cells, other methods for improving the stand capability of this system will have to be sought.

TABLE XIII
SELF-DISCHARGE IN CuF_2 -Li CELLS
 (Undried CuF_2)

Electrolyte: 15g LiClO_4 /100 ml PC

Cell No.	Stand Conditions		Theo. CuF_2 Cap., AH	Hrs. to 2.0VF	Capacity to 2.0VF, AH	Average Discharge Potential Volts	Cathode Eff., %
	Temp. °C	Time, wks.					
3A-1			3.74	135	1.88	2.77	50
3A-2			3.46	127	1.79	2.82	52
3A-3			3.78	131	1.83	2.79	49
3A-4			3.52	132	1.89	2.86	54
3A-5 ¹	+35	2	---	---	---	---	---
3A-6 ¹	+35	2	---	---	---	---	---
3A-7 ¹	+35	2	---	---	---	---	---
3A-8 ¹	+35	2	---	---	---	---	---
3A-9 ^{1,2}	+35	2	---	---	---	---	---
3A-10 ^{1,2}	+35	2	---	<6	---	---	---
3A-11	-15	2	3.64	42	0.55	2.61	15
3A-12	-15	2	4.04	64	0.85	2.65	21
3A-13	-15	4	4.66	49	0.49	2.76	11
3A-14	-15	4	4.03	43	0.56	2.62	14
3A-15 ¹	-15	6	4.18	---	---	---	---
3A-16 ¹	-15	6	3.69	<12	---	---	---
3A-17	-55	2	4.05	101	1.42	2.81	35
3A-18	-55	2	4.04	65	0.92	2.81	23
3A-19	-55	4	3.65	88	1.20	2.72	33
3A-20	-55	4	4.12	99	1.32	2.65	32
3A-21 ¹	-55	6	4.23	56	0.74	2.64	17
3A-22 ¹	-55	6	3.32	---	---	---	---

¹ Cell potential below 2 volts after stand

² Cell on 5,000 Ω load during stand

TABLE XIV
SELF-DISCHARGE IN CuF_2 -Li CELLS
 (Dried CuF_2)

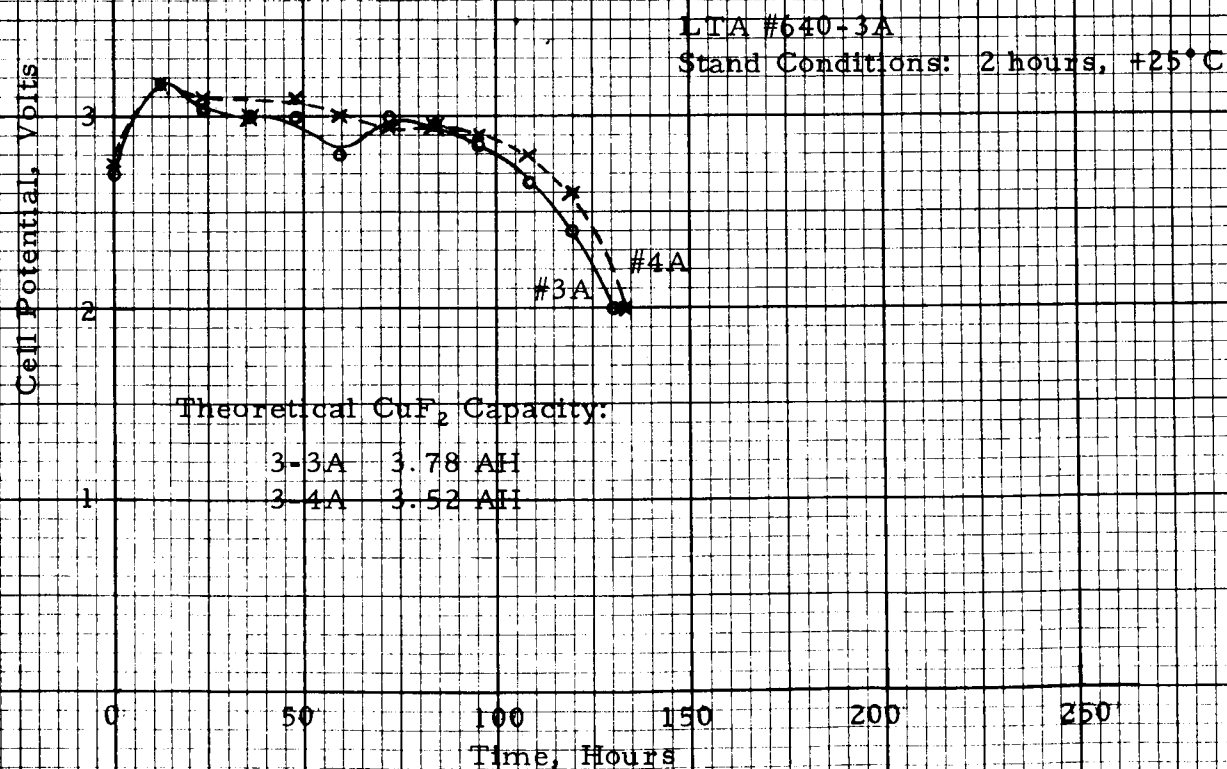
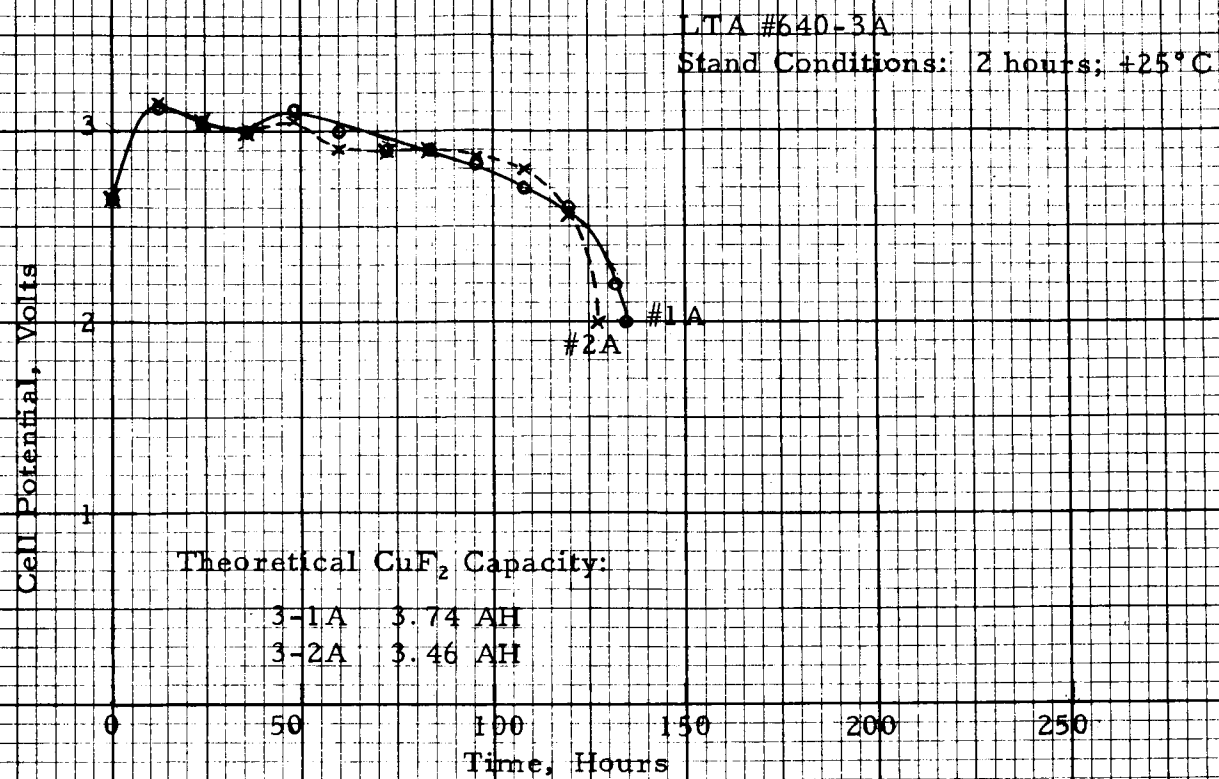
Electrolyte: 15g LiClO_4 /100 ml PC

Cell No.	Stand Conditions		Theo. CuF_2 Cap., AH	Hrs. to 2.0VF	Capacity to 2.0VF, AH	Average Discharge Potential, Volts	Cathode Eff., %
	Temp., °C	Time, wks.					
3-1			3.88	166	2.44	2.94	63
3-2			4.23	198	2.89	2.91	68
3-3			3.88	161	2.32	2.88	60
3-4			4.17	175	2.59	2.96	62
3-5 ¹	+35	2	---	---	---	---	--
3-6 ¹	+35	2	---	---	---	---	--
3-7	+35	2	3.82	33	0.41	2.45	11
3-8 ¹	+35	2	---	---	---	---	--
3-9 ^{1,2}	+35	2	---	---	---	---	--
3-10 ²	+35	2	3.98	24	0.28	2.34	7
3-11	-15	2	3.98	155	2.19	2.83	55
3-12	-15	2	3.74	147	2.07	2.82	55
3-13	-15	4	3.74	147	2.09	2.85	56
3-14	-15	4	3.84	164	2.38	2.90	62
3-15 ³	-15	6	3.64	72	1.01	2.80	28
3-16	-15	6	3.64	48	0.64	2.67	17
3-17	-55	2	3.53	101	1.41	2.79	40
3-18	-55	2	4.15	87	1.30	2.99	31
3-19	-55	4	3.87	148	2.09	2.81	54
3-20	-55	4	4.10	148	2.10	2.84	51
3-21	-55	6	4.33	168	2.37	2.82	55
3-22	-55	6	4.00	185	2.65	2.86	61

¹ Cell potential below 2 volts after stand

² Cell on 5,000 Ω load during stand

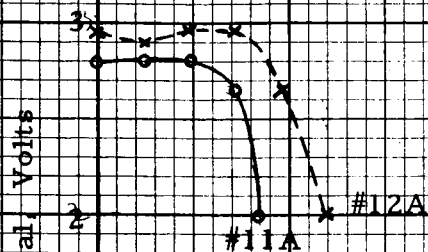
³ 2.52 VF



SELF-DISCHARGE IN CuF_2 -Li CELLS

FIGURE 26

LTA #640-3A

Stand Conditions: 2 weeks; -15°C Theoretical CuF_2 Capacity:

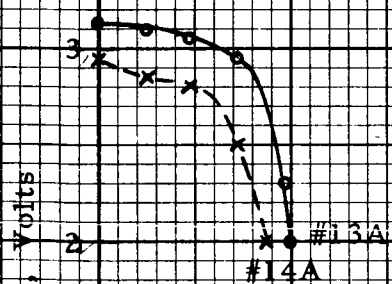
3-11A 3.64 AH

3-12A 4.04 AH

0 50 100 150 200 250

Time, Hours

LTA #640-3A

Stand Conditions: 4 weeks; -15°C Theoretical CuF_2 Capacity:

3-13A 4.66 AH

3-14A 4.03 AH

0 50 100 150 200 250

Time, Hours

SELF-DISCHARGE IN CuF_2 -Li CELLS
FIGURE 27

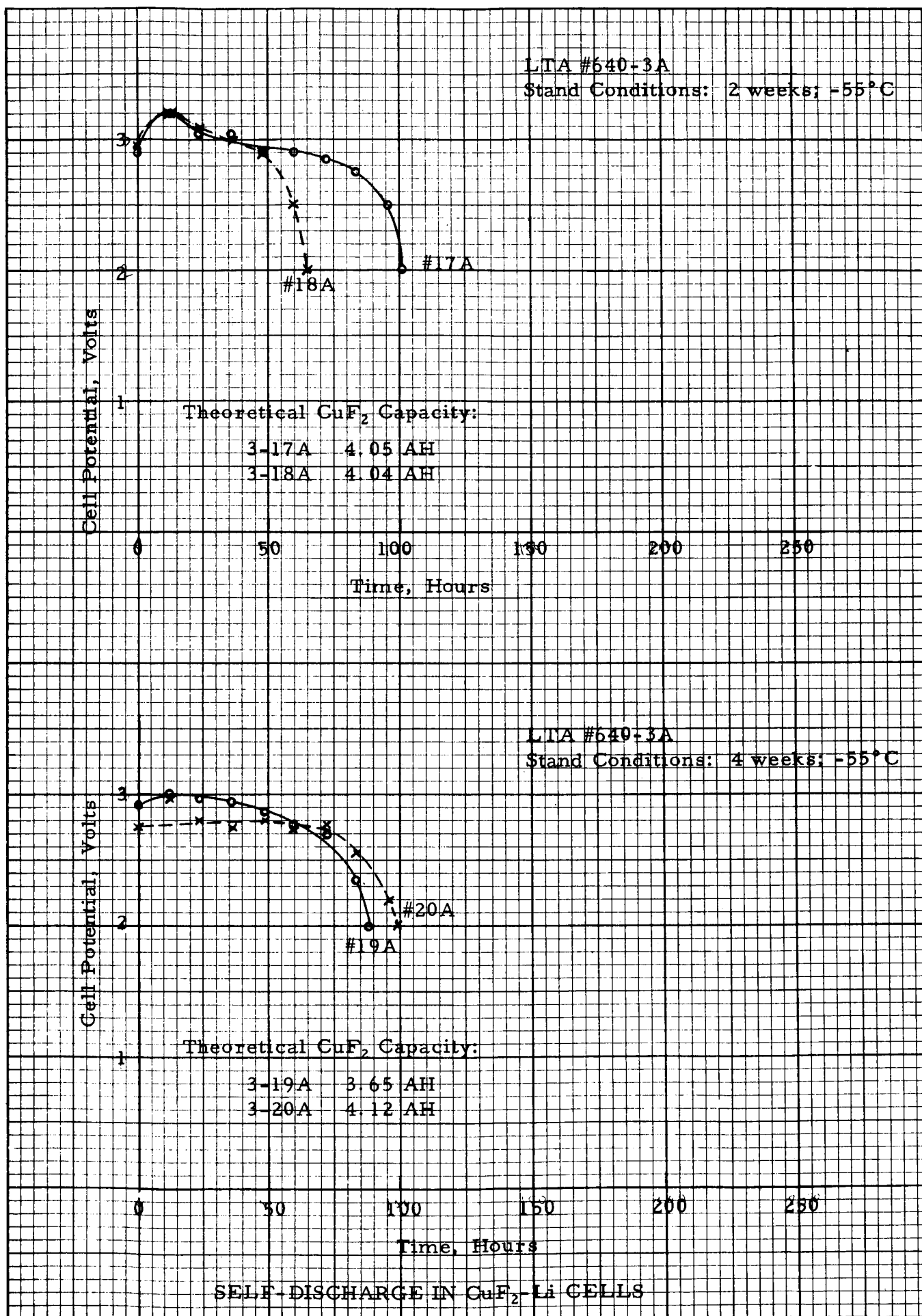
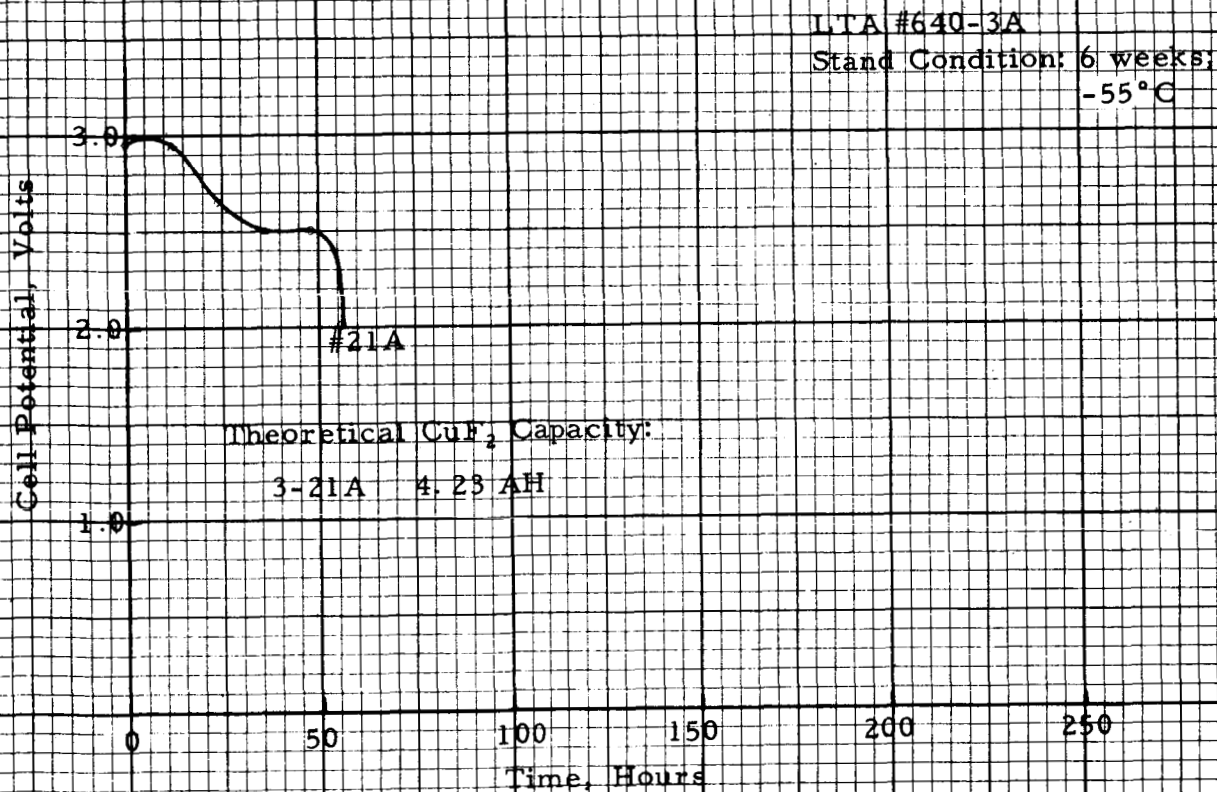


FIGURE 28

LTA #640-3A
Stand Conditions: 6 weeks; -15°C
Both cells below 2V after stand



SELF-DISCHARGE IN CuF_2 -Li CELLS
FIGURE 29

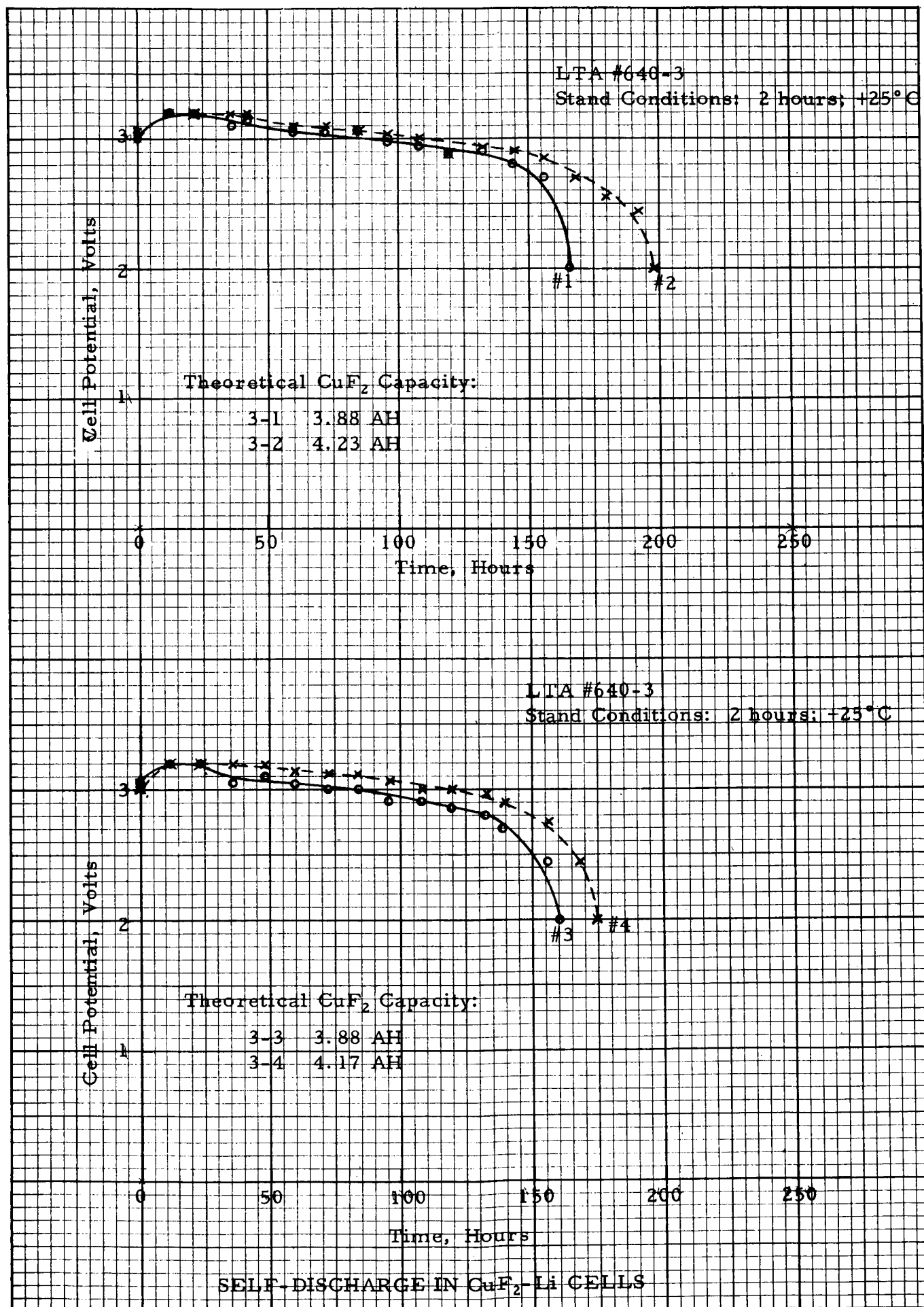


FIGURE 30

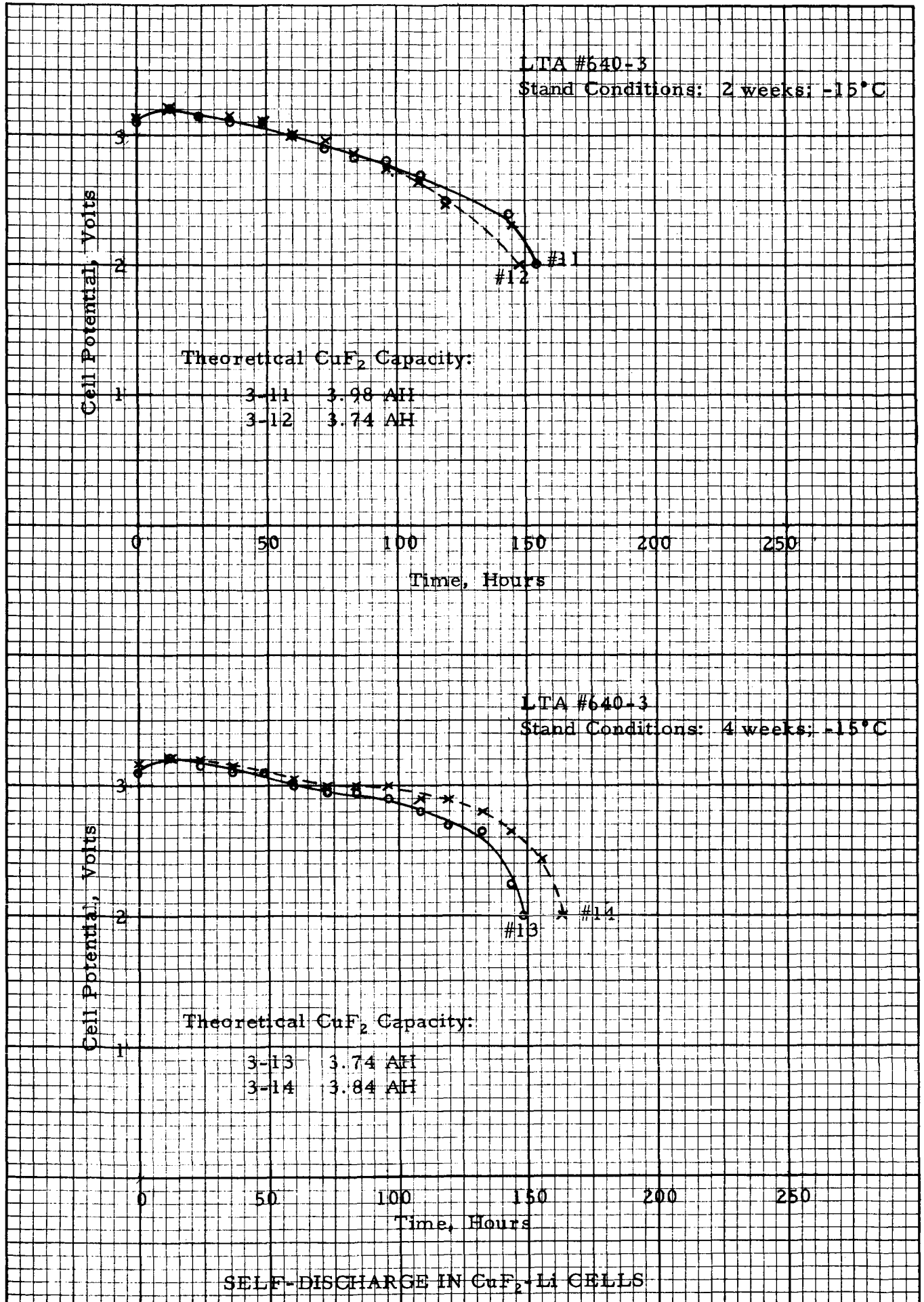


FIGURE 31

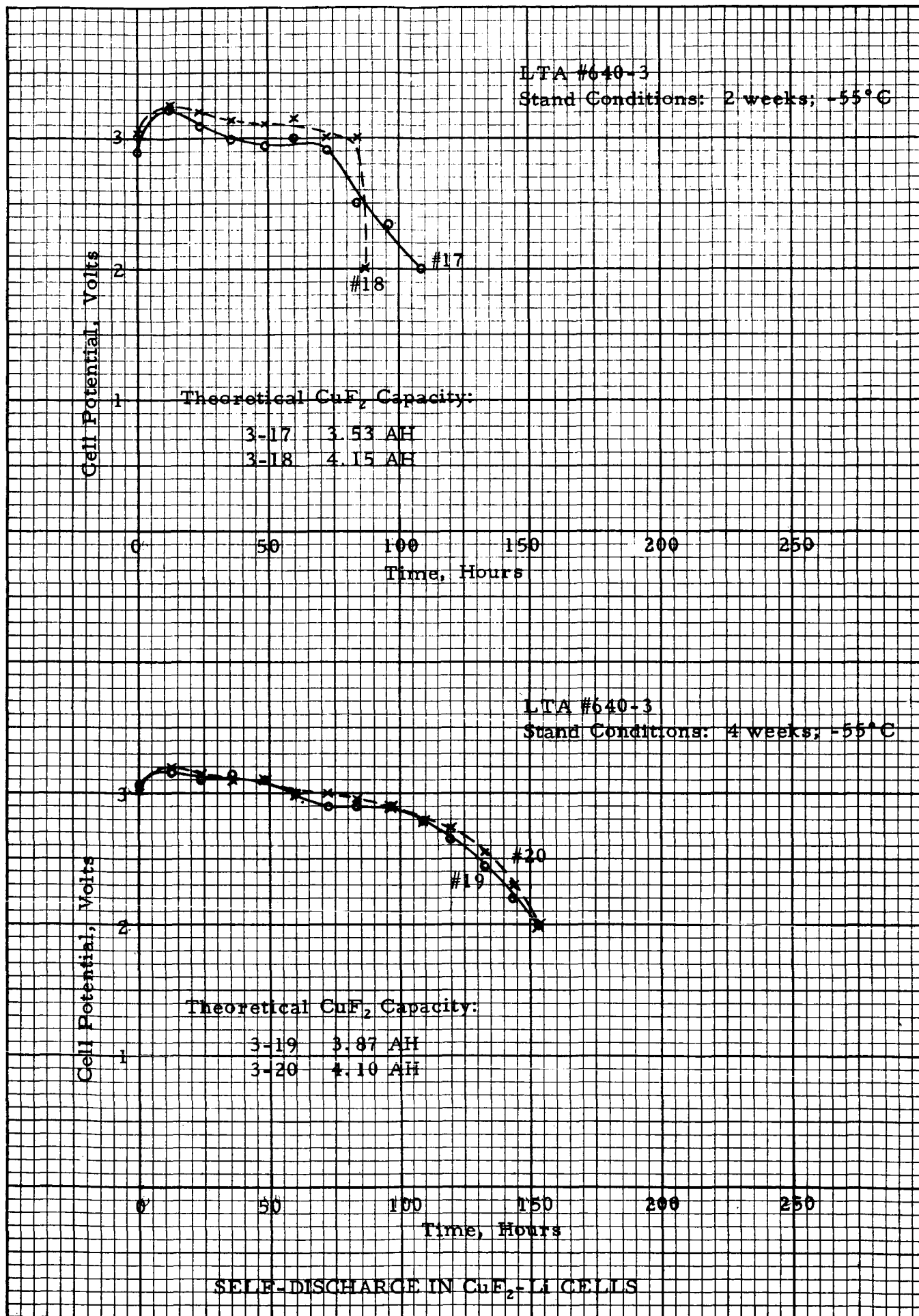


FIGURE 32

4. APPENDIX

DETERMINATION OF WATER IN COPPER FLUORIDE BY X-RAY DIFFRACTION

A quantitative determination of water in copper fluoride is obtained by measuring the quantity of $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$. Assuming that all water occurring in the copper fluoride combines with CuF_2 to form $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$,

The quantity of water present is given by the formula:

$$\begin{aligned} \% \text{H}_2\text{O} &= (0.262)(\% \text{CuF}_2 \cdot 2\text{H}_2\text{O in sample}) \\ &= \frac{(0.262) I_{\text{sample}} (\text{CuF}_2 \cdot 2\text{H}_2\text{O})}{I_{\text{pure}} (\text{CuF}_2 \cdot 2\text{H}_2\text{O})} \end{aligned}$$

where (0.262) is the stoichiometric fraction of water in $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$; $I_{\text{sample}} (\text{CuF}_2 \cdot 2\text{H}_2\text{O})$ denotes the intensity of the primary peak of $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ in the sample and $I_{\text{pure}} (\text{CuF}_2 \cdot 2\text{H}_2\text{O})$ denotes the intensity of the same peak in reagent $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$.

Copper fluoride samples to be analyzed are ground to give greater sample uniformity and sealed in polyethylene bags until ready to be run.

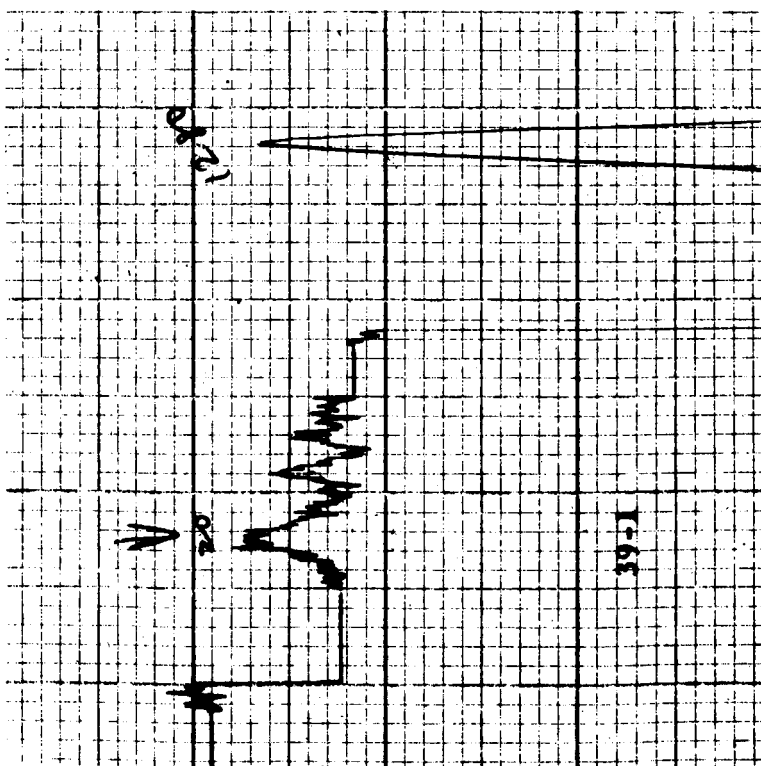
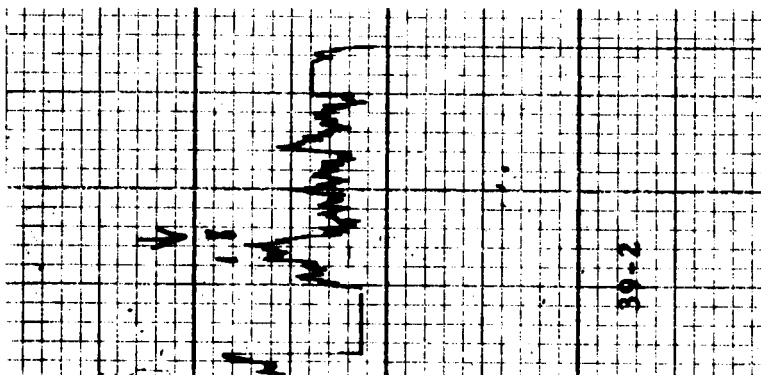
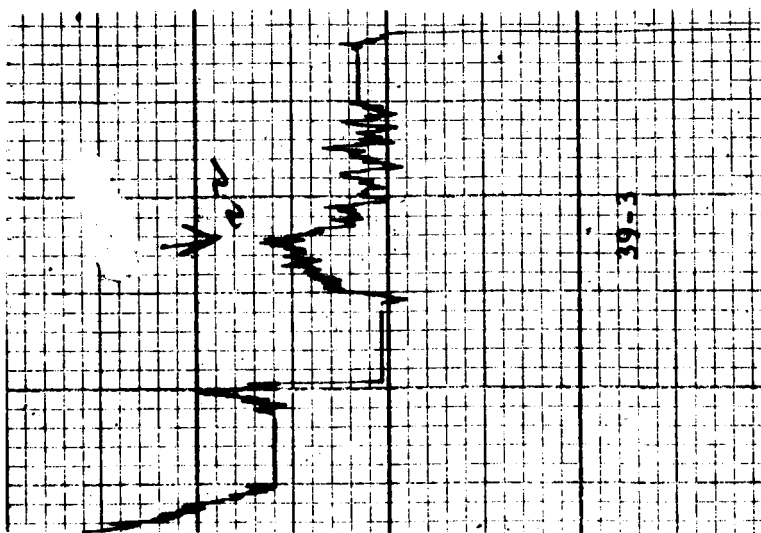
Prior to the diffraction analysis the powder sample is pressed into a die and a level surface is scraped clean. The sample thus prepared is run at $1^\circ/\text{min}$ on a Phillips diffractometer and intensities are recorded at the primary peaks of $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ and CuF_2 , the latter giving an indication of the relative purity of the copper fluoride sample.

To determine the consistency and reliability of the water determination, triplicate samples were analyzed. The variation among the tests depended upon the concentration of the dihydrate in the sample and ranged from a maximum of 5 percent for concentrations from 2 percent to 100 percent, to as much as 20 percent for concentrations in the neighborhood of 1/2 percent to 1 percent. Figures 33, 34, and 35 show tests on samples having less than .3 percent water. The following table shows the respective intensity measurements and the reported water levels for these materials.

<u>33 - 5, 6, 7</u> <0.1% H_2O	<u>33 - 8, 9, 10</u> <0.2% H_2O	<u>39 - 1, 2, 3</u> <0.3% H_2O
12	12	22
12	12	18
16	8	20

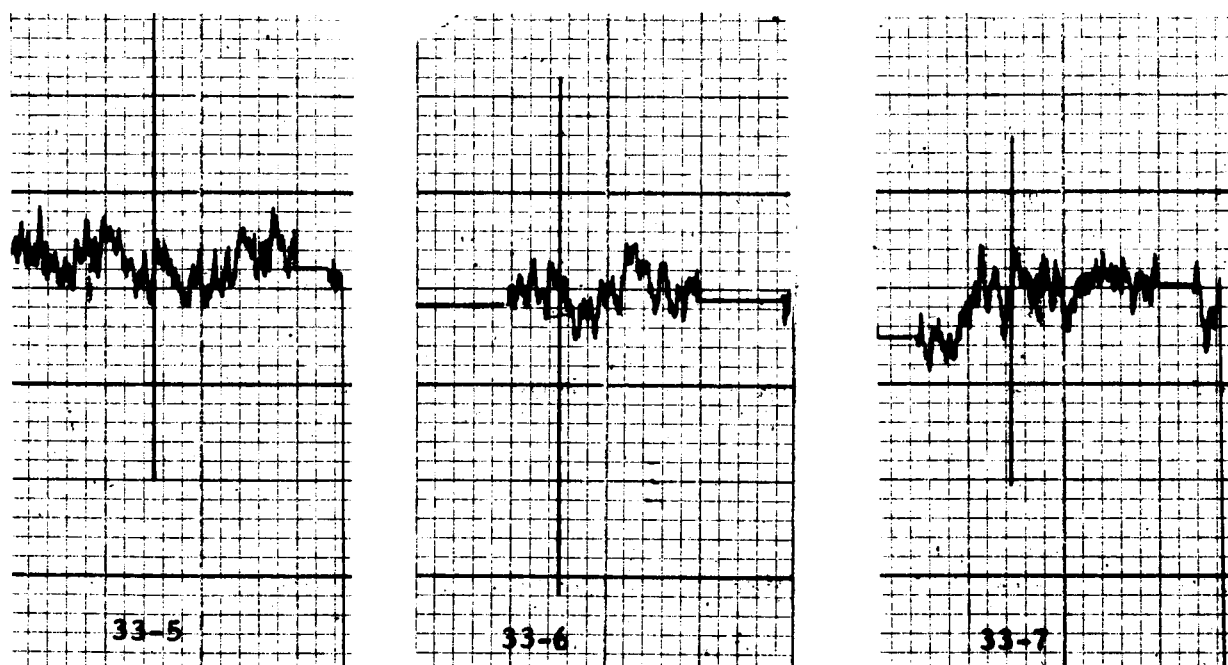
For $\%H_2O < 0.3\%$ only a maximum on the water level can be set, whereas above this a definite $\%H_2O$ can be obtained. Sample No. 39-1, 2, 3 above is a borderline case and, as can be seen in Figure 33, has a clear peak whereas the other samples do not have such a peak.

To determine the reliability of the results, samples prepared by mixing known quantities of CuF_2 and $CuF_2 \cdot 2H_2O$ were analyzed. The results are shown on Table XV, with accompanying graphs in Figures 36, 37, 38, and 39. Agreement between the X-ray and calculated figures is quite satisfactory over the sensitivity range of the test.



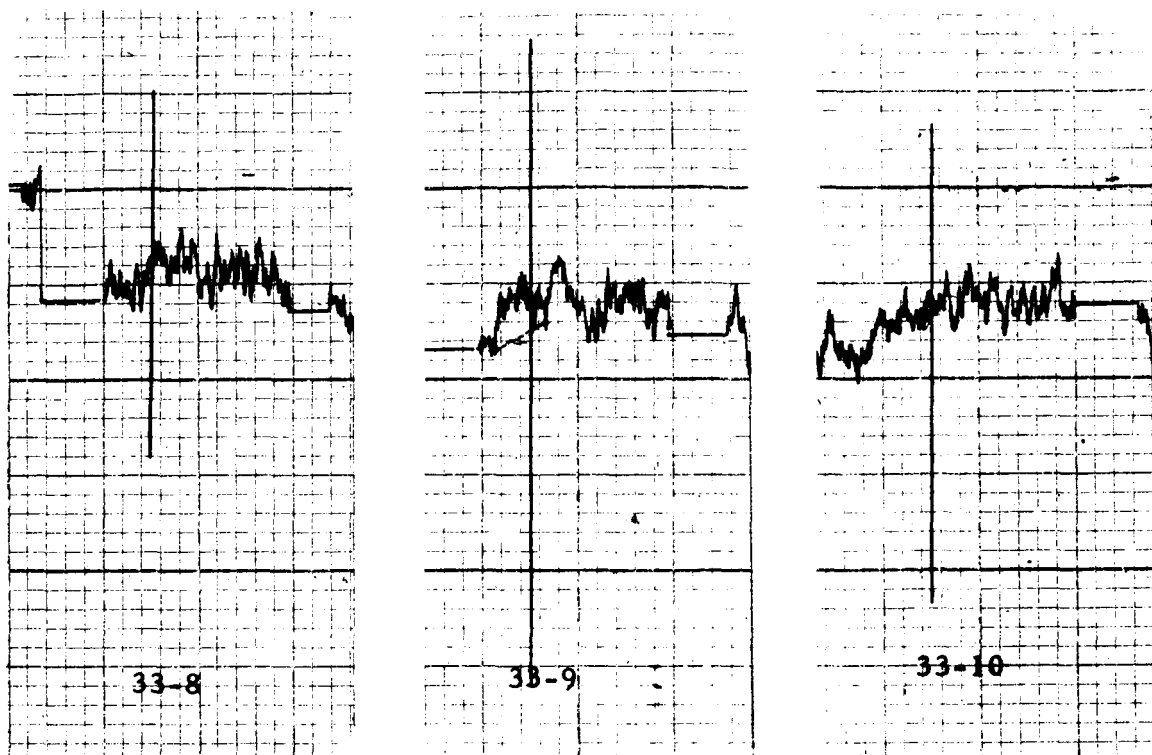
0.3% H₂O

FIGURE 33



0.1% H₂O

FIGURE 34



0.2% H_2O

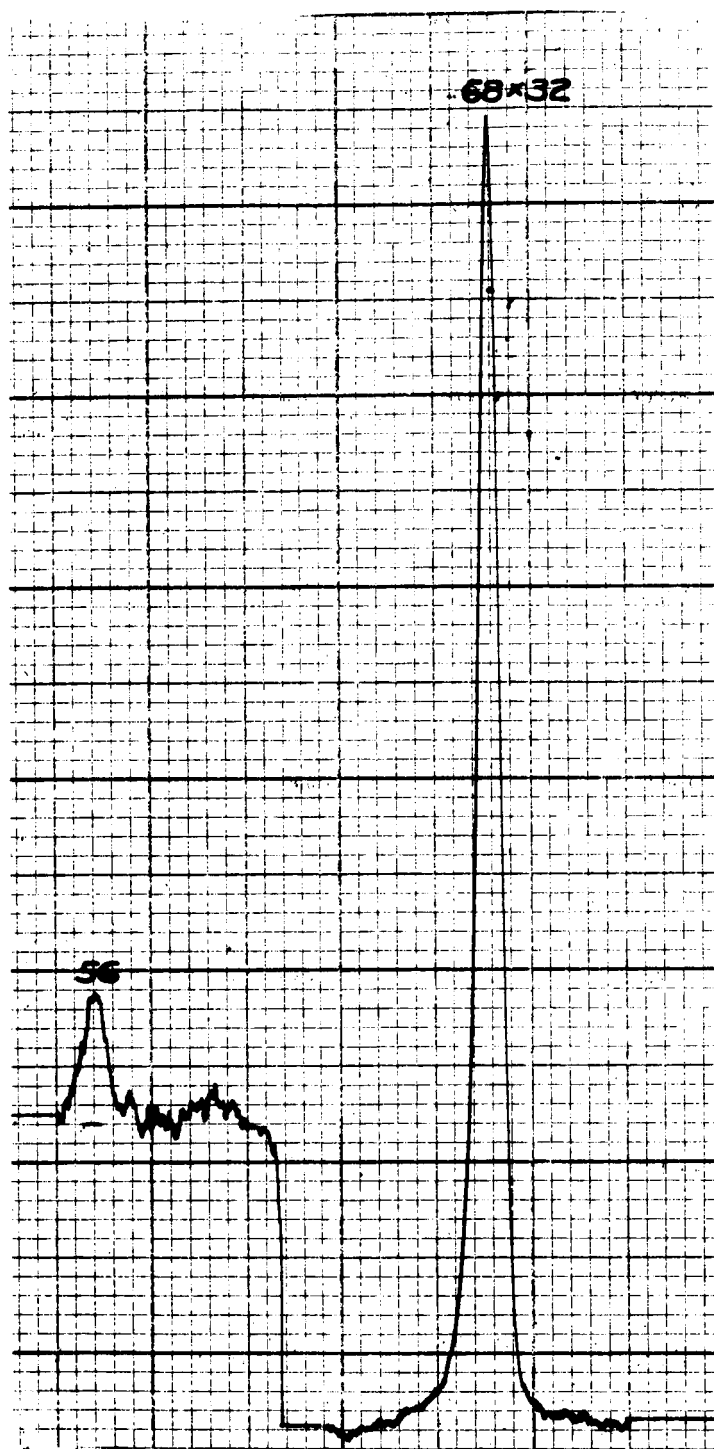
FIGURE 35

TABLE XV

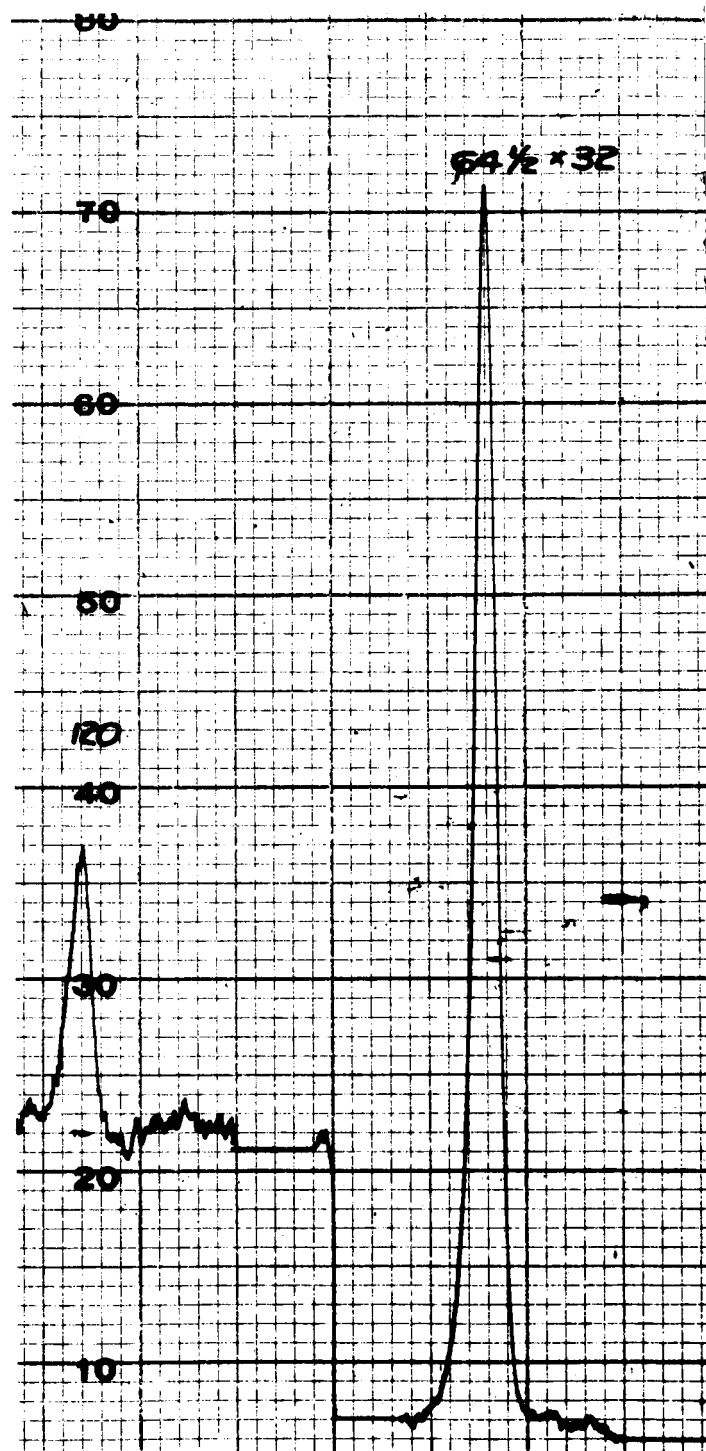
X-RAY DIFFRACTION STANDARDS

grams $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ per 100 g sample		gms. CuF_2 Lot 5 #1 per 100 g sample	gms. H_2O per 100 g sample due to $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$	gms. H_2O per 100 g sample due to Lot 5	Calculated gms. H_2O per 100 g sample	gms. H_2O per 100 g sample by X-ray
Actual X-ray Anal- ysis						
100	100	0	26.2	0	26.2	Std.
80	85	20	21.0	0.03	21.0	22
50	50	50	13.1	0.07	13.2	13
20	19	80	5.2	0.11	5.3	4.9
10	9.5	90	2.6	0.13	2.7	2.5
5	5.5	95	1.3	0.13	1.4	1.4
2	2.6	98	0.5	0.14	0.64	0.67

Note: Water content in Lot 5 $\text{CuF}_2 = 0.14\%$ (X-ray).

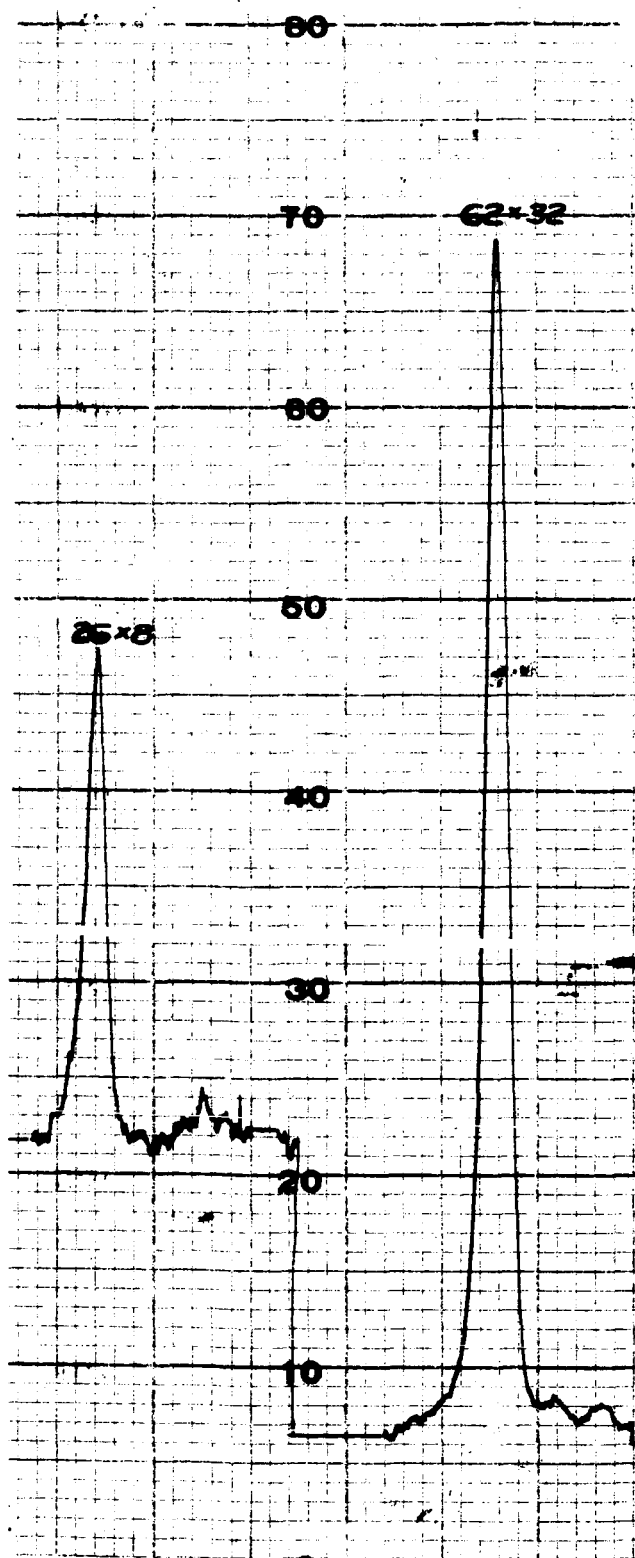


2 g $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ per 100 g of Sample

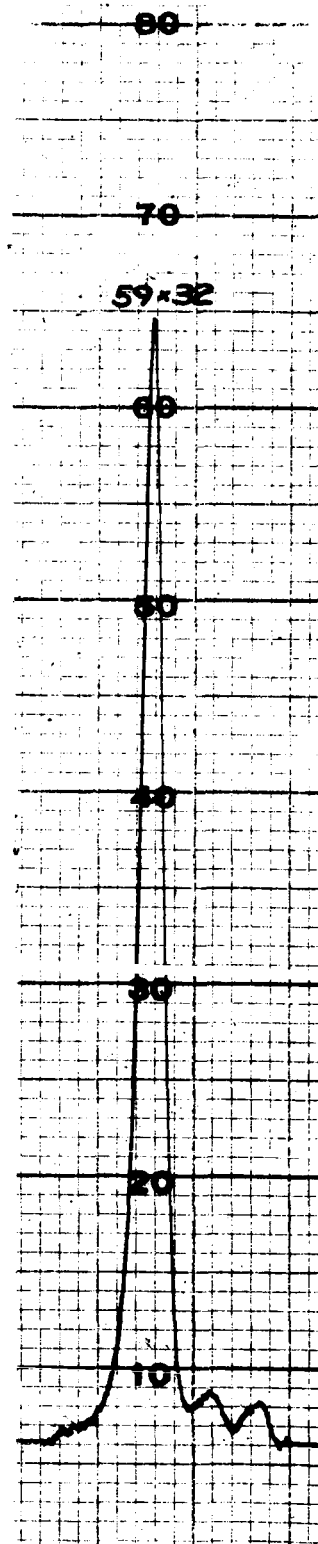
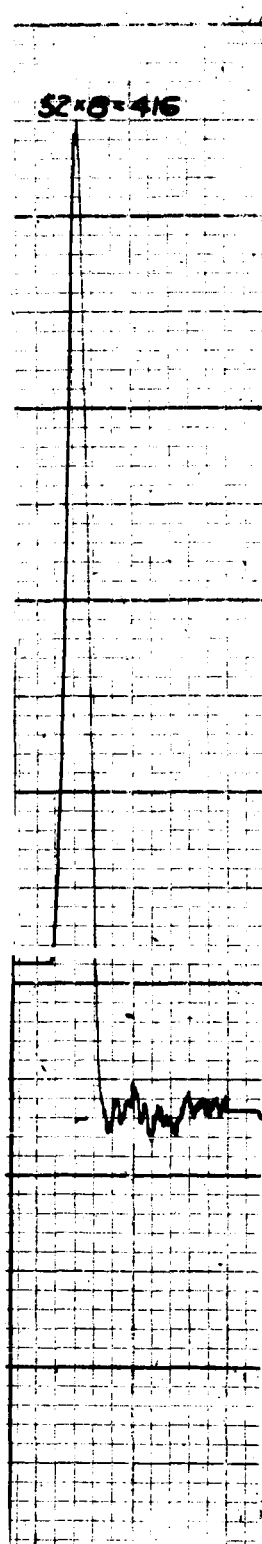


5 g $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ per 100 g of Sample

FIGURE 36



10 g $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ per 100 g of Sample



20 g $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$
per 100 g of Sample

FIGURE 37

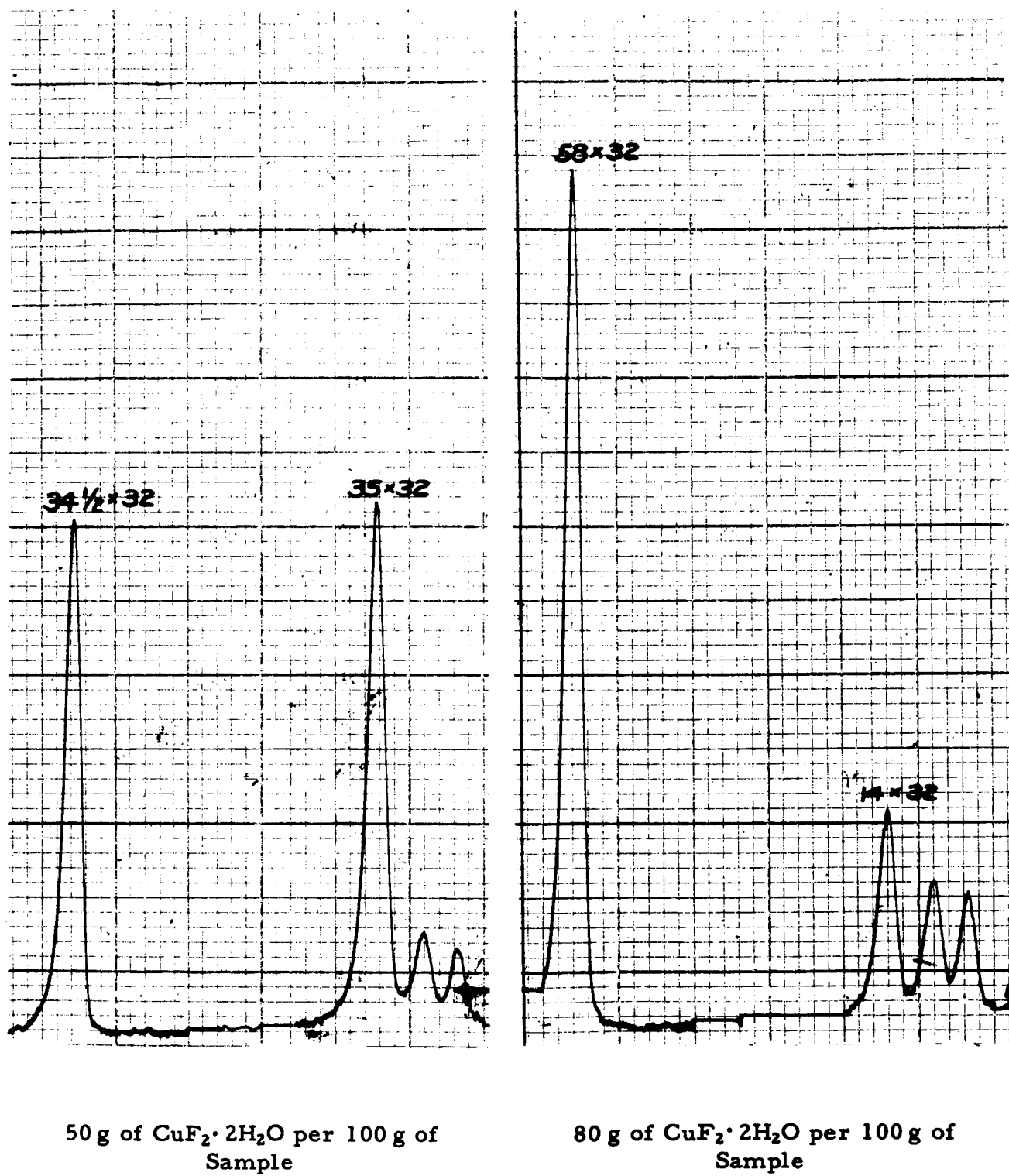
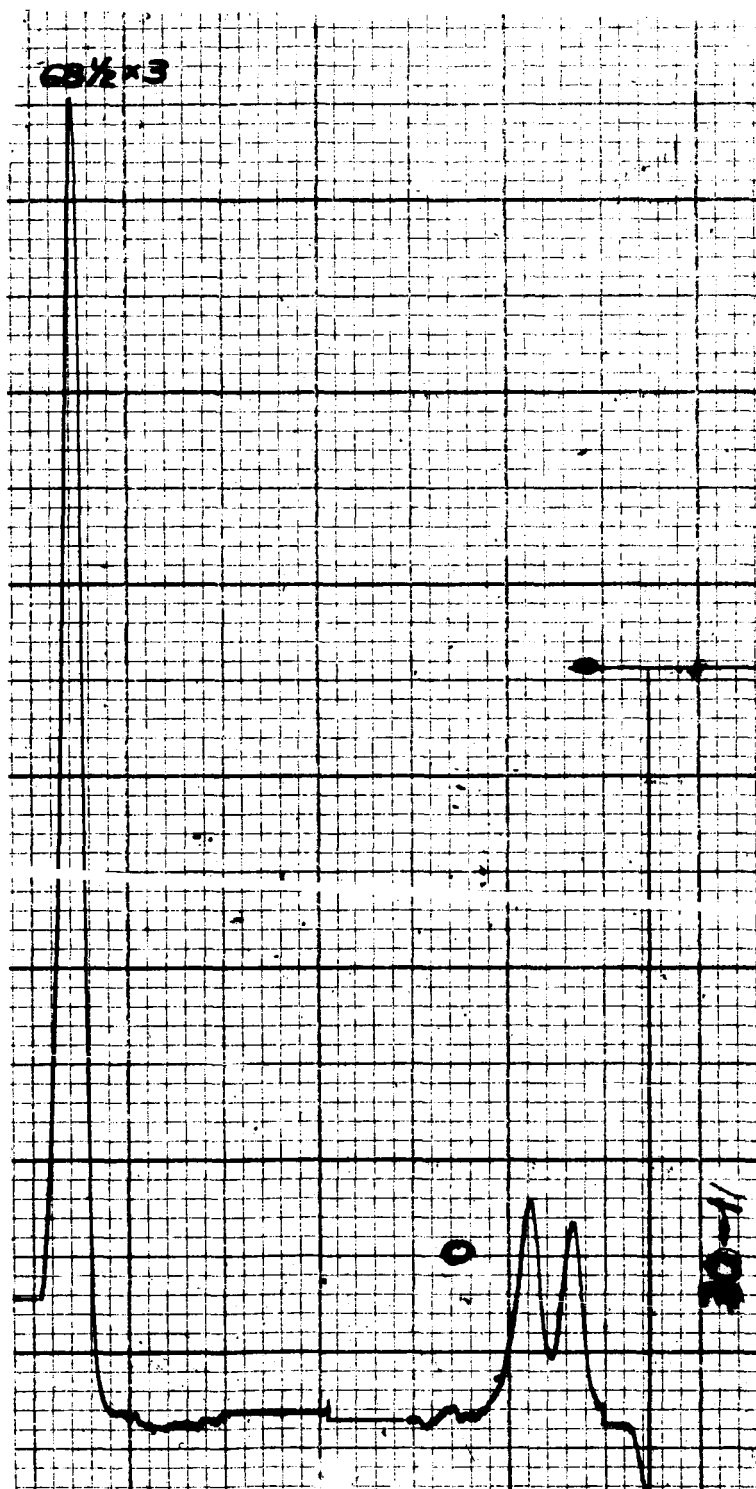


FIGURE 38



100 g of $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ per 100 g of Sample

FIGURE 39

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Joseph Sherfey, Code 735
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